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EFFECT OF OVERHEATING ON CREEP-RUPTURE PROPERTIES OF S-816 ALLOY AT 1,500° F

By John P. Rowe and J. W. Freeman

University of Michigan

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SUMMARY

An investigation of overheating S-816 alloy to temperatures of 1,650°, 1,800°, 1,900°, and 2,000° F during the course of rupture tests at 1,500° F was carried out. The overheating was applied periodically for 2 minutes in most of the tests. The intent was to develop basic information on the effect of overheats on creep-rupture properties in order to assist in the evaluation of damage from overheats during gas-turbine operation.

Overheating reduces rupture life both through alteration of the internal structure of the alloy and, if stress is present during an overheat, by accelerated creep at the higher temperature. Such reduction in rupture life increases with the temperature and duration of overheating. Loss in rupture life by structural alteration was negligible at 1,650° F, but two overheats to 2,000° F of 2-minute duration in the absence of stress reduced life at 1,500° F by about 70 percent. Apparently, the total damage, if stress is present during overheats, is the sum of the structural change effect from temperature plus the percentage of the total rupture life at the overheat temperature represented by the time at the overheat temperature under stress.

While the reduction in rupture time at 1,500° F due to temperature-induced structural changes can be large, the corresponding reduction in stress for rupture in a specific time is considerably smaller on a percentage basis. From this viewpoint, major reductions in rupture strength due to overheating arise only when sufficient stress is present during an overheat to use up substantial amounts of rupture life by accelerated creep. This indicates that in the absence of substantial creep during overheating other sources of damage, such as thermal shock, will usually be the important causes of damage.

INTRODUCTION

An investigation was carried out to evaluate the effects of brief overheats to temperatures of 1,650°, 1,800°, 1,900°, and 2,000° F on the creep-rupture properties of S-816 alloy at 1,500° F. The objective of the investigation was to obtain basic information on the changes in creep-rupture properties of the alloy due to overheating which can occur during jet-engine operation.

The effects of overheating were evaluated in terms of the changes in creep-rupture characteristics at 1,500° F under stresses within the range of rupture strengths of the S-816 alloy for 100 to 1,000 hours. The possible damage from overheating was considered to include internal metal structure changes induced by exposure to the higher temperatures and loss in life by creep if stress was present during the overheats. Temperature damage was evaluated by starting tests at 1,500° F and then periodically overheating with the stress removed during the overheat periods. Stress damage during overheats was evaluated by leaving stress on the specimens during the overheats.

Overheat periods were predominately 2 minutes in duration and were applied cyclically at approximately 5- or 12-hour intervals. These schedules were adopted to provide the most useful general results after consideration by the Subcommittee on Power-Plant Materials of the National Advisory Committee for Aeronautics of the variable conditions under which overheating can occur in jet-engine service. It should be clearly recognized that the intent was to develop general principles and not to evaluate the specific conditions of overheating which can occur in a specific jet engine. The investigation was also limited to the effects on creep-rupture properties. The overheat conditions did not include the effects of differential restrained expansion (thermal shock) or the possible effects on such other properties as fatigue strength and corrosion resistance.

This investigation was carried out by the Engineering Research Institute of the University of Michigan under the sponsorship and with the financial assistance of the NACA. It was part of a general research investigation studying metallurgical factors involved in the use of heat-resistant alloys in aircraft propulsion systems.

PROCEDURE

Overheating can be expected to have two main effects on creep-rupture life at some lower nominal temperature:

(1) Change of creep-rupture life due to the exposure to a higher temperature changing the internal structure of the metal. This effect is designated "temperature damage" in subsequent discussions.

(2) Acceleration of creep when the temperature is increased in the presence of stress, subsequently referred to as "stress damage."

In addition, the cyclic removal and reapplication of the stress during the overheat experiments in the absence of stress could alter the creep-rupture characteristics. The influence of overheats could also be expected to vary depending on the stress level and rupture time at the nominal test temperature. In consideration of these factors, the following general experimental program was established.

Determination of Temperature Damage From Overheating in Absence of Stress

The following data were obtained in the determination of temperature damage from overheating in the absence of stress:

(1) The basic measurement was the effect on rupture time of continuous cyclic overheats to 1,650°, 1,800°, 1,900°, and 2,000° F until rupture occurred. For tests under a 16,200-psi stress at 1,500° F (rupture in 1,210 hours) the load was removed and an overheat applied twice daily. For tests under a 22,000-psi stress at 1,500° F (rupture in 94 hours) the overheats were applied every 5 hours.

(2) The accumulative damage of overheats was established by stopping the overheats after various amounts of the total rupture life had been used up. The tests were then allowed to proceed to rupture at a constant load at 1,500° F. This procedure enabled the development of curves of percent of the nominal life at 1,500° F against the amount of overheating.

(3) The relation of the occurrence of overheating to the degree to which creep had proceeded at 1,500° F was checked by delaying overheats until the second stage of creep. This provides information as to whether there is a difference in the damage from overheats depending on the degree of prior service.

As part of this procedure, specimens were overheated before starting the rupture tests at 1,500° F. This type of test would primarily indicate whether such experiments provide reliable data regarding the effects of overheats during service.

(4) Tests were also carried out in which the load was removed and reapplied as in the overheat cycles without changing the temperature. These tests provide data as to the possible influence of cycling the load alone.

In addition to rupture-time changes, data were also obtained for the influence of the overheats on elongation and on the creep characteristics in the rupture tests.

Overheating in Presence of Stress to Establish

Stress Damage

Consideration of the characteristics of stress and rupture time at the overheat temperatures indicated that the total rupture times under the basic test stresses would be as follows:

Stress, psi	Normal rupture time at 1,500° F, hr	Rupture time, hr, at overheat temperature of -			
		1,650° F	1,800° F	1,900° F	2,000° F
12,500	>10,000	150	3.1	0.25	0.04
16,200	1,210	27	.65	.067	[a]>T
18,000	500	14	.3	[a]>T	[a]>T
22,000	94	3.8	[a]At T	[a]>T	[a]>T

[a]T, tensile strength.

These data immediately make it clear that overheating to 1,800° F or above under the stresses used for rupture at 1,500° F in time periods up to 1,000 hours would use up a very large proportion of the available rupture life. In other words, the time under stress at the overheat temperature would largely govern rupture life. For instance, the rupture time at 1,900° F under 16,200 psi is only 4 minutes. One 2-minute overheat to 1,900° F under this stress would use up half of the available rupture life. Obviously, 16,200 psi is above the ultimate tensile strength at 2,000° F and 18,000 and 22,000 psi are above it at both 1,900° and 2,000° F.

Consideration of the problem involved led to a restricted test program designed to determine if addition of the temperature damage together with the percentage of rupture life used up during the time at the overheat temperature could be used to predict rupture time. In order to reduce the testing time as much as possible, it was decided to use 18,000 psi, the stress at 1,500° F causing rupture in 500 hours. Any higher stress would have so shortened the normal rupture time that it would be difficult to develop significant data.

A schedule of overheating twice each day for 2 minutes was adopted. For tests with an overheat temperature of 1,650° F, it was decided to leave the stress at 18,000 psi and determine the number of overheats to cause fracture. Even if the test lasted 500 hours the total time at 1,650° F would be only 84 minutes in comparison with the total rupture time of 14 hours at 1,650° F for an 18,000-psi stress.

For overheating at 1,800° F, however, it was necessary to reduce the stress in order to restrict the damage from creep at 1,800° F to a level where the temperature damage would also be significant. The procedure of reducing the stress to 12,500 psi during overheats was adopted using the following reasoning:

(a) The time and stress at 1,800° F were selected to use up 30 percent of the total rupture time at 1,800° F. This then ought to reduce the rupture time to 350 hours at 1,500° F in the absence of any other effect.

(b) There would be 29 overheats in 350 hours with a total time at 1,800° F of 58 minutes.

(c) The stress causing rupture in 58/0.3 = 193 minutes at 1,800° F was 12,500 psi. (See table I.)

The curves of stress against rupture time for tests at constant loads and constant temperatures were all established by the usual practice of bringing the specimen to temperature in the furnace, adjusting the temperature, and then loading. This involved several hours of heating prior to loading. A few tests were run in which the temperature was attained by resistance heating to see if this altered the short-time, high-temperature, rupture characteristics.

MATERIAL

The material for this investigation was 7/8-inch bar stock from commercial heat A6507 made by the Universal Cyclops Steel Corp. and supplied gratis by the Thomson Laboratory of the General Electric Co. Its analysis was reported to be as follows:

Chemical composition, weight percent									
C	Mn	Si	Cr	Ni	Co	Mo	W	Cb	Fe
0.42	1.37	0.58	20.00	20.40	Balance	3.93	3.57	3.50	3.90

Initially, it was found that the standard heat treatment (1 hour at 2,150° F then water-quenched plus 12 hours at 1,400° F and air-cooled) on this stock produced nonuniform grain size known to cause unpredictably variable rupture properties. To avoid this difficulty and to produce more uniform material, the 7/8-inch bar stock was hot-rolled to 1/2-inch broken-cornered squares at the University of Michigan. The rolling was done from 2,150° F with one reheat. This material had the following average rupture strengths in comparison with published values for the alloy:

Time for rupture, hr	Strength of test stock, psi	Range of strengths reported (ref. 1), psi
100	21,500	19,000 to 26,000
1,000	16,500	15,000 to 22,000

EXPERIMENTAL TECHNIQUES

The conduct of such an investigation required the modification of existing equipment and techniques to include the flexibility necessary for the many aspects considered.

Testing Equipment

The creep-rupture testing was carried out in conventional beam-loaded creep-rupture units using specimens with a 0.250-inch diameter and 1-inch gage length. Each sample was accurately measured before testing. Time-elongation data were taken during the tests by the use of a modified Martens-type optical extensometer with a sensitivity of ±0.00001 inch. The units were equipped with automatically controlled resistance furnaces. Temperature variations along the gage length were held to ±3° F. For all tests the furnaces were turned on and allowed to come to temperature overnight. The specimens were then placed in the hot furnace, brought on temperature, and loaded in a maximum of 4 hours.

For overheat tests, the conventional units were modified to permit resistance heating of the specimens by passing heavy direct current through the sample. A 400-ampere, direct-current generator was used as a power supply. In order to avoid disturbing the specimen during the test, insulated terminal blocks were fastened to the frame of the unit level with the top and bottom of the furnace. From these terminals short leads were fastened to the top and bottom specimen holders before the test was started. Then, for overheating, it was necessary to attach the power supply leads to only the terminal blocks, completing the circuit to a generator field switch. The top specimen holder was insulated from the frame by means of a Transite insert. The whole circuit was grounded either through the beam or through an attached ground wire. A photograph of a unit is shown as figure 1.

In order to follow the temperature accurately during an overheat, a welding technique (ref. 2) was employed using Chromel-Alumel thermocouples and an electronic indicating potentiometer. A schematic sketch of this arrangement is shown as figure 2. A problem stemmed from two factors. In order to follow the rapidly changing temperatures during an overheat cycle and effect accurate control, the thermocouple wires had to be welded to the sample. This was done with a percussion-type welder. The welded attachment maintained the thermocouple bead in contact with the specimen as reduction in cross section occurred by creep during the tests. In welding the thermocouple wires on the specimen, however, any minute error in positioning either wire caused the direct current from the generator to impress an electromotive force on the thermocouple circuit. This electromotive force varied with the magnitude of the placement error and appeared on the temperature indicator as a temperature effect. To avoid this, two Alumel wires were employed, one deliberately placed on either side of the single Chromel wire. By connecting these two Alumel wires to the extremes of a variable resistance, the variable tap could be adjusted so that the two electromotive forces obtained cancelled each other, leaving only the thermal electromotive force impressed on the indicator.

Checks were made of the original calibration and the maintenance of calibration of the thermocouples. The system used gave accurate temperature measurements as installed. The cyclic overheats did not change the calibration by any more than 1° F at any of the temperatures.

Overheating Procedures

This investigation included three types of overheats: Overheats before testing, overheats in the absence of stress, and overheats in the presence of stress. Each type required a different procedure involving the equipment described above.

Overheats before testing.- Overheating before testing was done in two ways depending on the duration and temperature of overheating. The procedures used were as follows:

(1) Tests of specimens overheated to 1,600° F for long time periods were loaded in the creep furnace exactly as for a creep-rupture test. After being brought on temperature at 1,500° F, the furnace temperature was raised rapidly to 1,600° F, held for the desired time period, and then cooled to 1,500° F. The load was then applied and the test run to rupture.

(2) Samples overheated to 1,600° F for short time periods and all samples overheated to 1,800° and 2,000° F before testing were prepared in the following manner. A thermocouple was attached to each sample. A heat-treating furnace was brought on temperature and held to assure equilibrium. The samples were then placed in the furnace and the time counted from the point at which the temperature indicated by the attached thermocouple reached 10° F below the equilibrium furnace temperature. Following completion of the desired time at temperature, the specimens were removed from the furnace and air-cooled. They were then set up and the test run as a standard creep-rupture test.

Overheats in absence of stress.- All overheating done in the absence of stress was of a cyclic nature where the described cycle was repeated a predetermined number of times. For these tests the specimens were prepared with a thermocouple welded at the center as described previously and an additional thermocouple mechanically attached at each end of the reduced section for checks on temperature distribution along the gage length. They were placed in the creep furnace and started exactly as in a normal creep-rupture test except that the short power leads were attached to the specimen holders before stressing. Then, after the completion of the desired time period before the first overheat, the following procedure was followed in performing an overheat:

(1) The temperature was checked and an elongation reading was made. At this time, the power leads from the generator were attached to the unit and the welded thermocouple was connected to the indicating potentiometer.

(2) The load was removed.

(3) After a 60-second time lapse during which the furnace input was cut back and the thermocouple circuit checked, the heating cycle was initiated by applying the maximum generator output of 400 amperes to the specimen. When the desired overheat temperature was attained the generator output was cut back to a value just sufficient to maintain temperature.

(4) At the end of the established cycle duration the power supply was cut off and the specimen allowed to cool. No forced cooling was employed other than that supplied by having allowed the furnace temperature to fall below 1,500° F when the input was reduced in step (3).

(5) The load was reapplied when the temperature reached 1,510° F, since this time was more nearly constant than the time to reach 1,500° F, and the furnace input was manipulated to bring the temperature on at 1,500° F as soon as possible.

(6) When temperature equilibrium was reestablished at 1,500° F, elongation measurements were taken again and the test continued to the next cycle. In plotting the time-elongation data, this reading after reapplication of the load was assumed to be at the same total deformation as the reading taken just prior to removal of the load at the beginning of the cycle.

(7) Typical time-temperature changes for overheats of 2 minutes to each of the temperatures used are shown in figure 3.

Overheats in presence of stress.- With a few exceptions in technique, tests of specimens overheated in the presence of stress were performed exactly as were the ones where stress was absent during overheats. The only difference was the omission of the steps involving removal and reapplication of the load. Deformation measurements were made before each cycle and again after equilibrium was reestablished at 1,500° F to measure the deformation which occurred during each overheat cycle.

Metallurgical Studies

The chief tool used in evaluating the cause of the observed effects of overheating was microstructural examination of the test samples. Longitudinal sections of the fractured specimens were cut from the gage length at the fracture. These were mounted and mechanically polished after grinding the cut surfaces to remove any cold-work left by the cutoff operation. The polished surface was then etched by immersion for about 10 seconds in a mixture containing 92 percent hydrochloric acid, 5 percent sulfuric acid, and 3 percent nitric acid. Photomicrographs were taken at magnifications of 100 and 1,000 to show both the general overall structure and the details of the precipitates present in each sample.

RESULTS AND DISCUSSION

The test results definitely show that for S-816 alloy at 1,500° F there is a reduction in rupture life due to overheat-temperature effects as well as a loss in life due to the presence of stress during overheating.

Rupture Properties of Test Material

All evaluations of overheat effects had to be based on changes in rupture time. The basic rupture-time data for tests without overheats are given in table I and shown as curves of stress against rupture time by figures 4 and 5. The objective of these tests was to establish rupture times for the various stresses used in the investigation.

A number of tests was carried out at 1,500° F to obtain an indication of the changes in rupture times which would be significant. The range in rupture times (fig. 5) was from 75 to 115 percent of the average rupture life at either 22,000 or 16,200 psi. Thus, for a measured change in rupture life from overheating to be significant, it has to be at least more than this range.

There was also need to know the rupture time under the stresses of interest at the overheat temperatures. The tests conducted gave the results shown by table I and figure 4. The main use of these data was to establish the relation of the time under stress at the overheat temperature to the time for rupture for the stress. In general, the scatter in the rupture times at these higher temperatures was not well established.

The original intent was to carry out tests in which all stress was removed during the overheat cycle, using stresses at 1,500° F which normally cause rupture in 100 and 1,000 hours. The stresses actually used, 22,000 and 16,200 psi, were selected before the range in properties was available. Consequently, the average rupture times turned out to be slightly different from 100 and 1,000 hours.

Overheating in Absence of Stress

Overheats were conducted in the absence of stress to 1,650°, 1,800°, 1,900°, and 2,000° F. Stresses of both 16,200 (stress for rupture in 1,210 hours) and 22,000 psi (stress for rupture in 94 hours) were used at 1,500° F. In most of the tests, the load was removed and the specimens overheated for 2 minutes, cooled back to 1,500° F, and reloaded every 12 hours for the tests under 16,200 psi. For the 22,000-psi tests, the overheats were applied every 5 hours. Tests were also carried out for material overheated before testing. Load cycling without temperature change at 1,500° F was also studied to learn how much effect the removal and reapplication of the load had on rupture time.

In addition to the changes in rupture time due to overheating, information was obtained on its effect on elongation in the rupture tests, effect on creep curves, and effect on the time to reach a given total deformation.

Effect on rupture life at 1,500° F of overheating in absence of stress.- The data (table II and figs. 6 to 8) clearly show that when the stress is removed from the S-816 alloy during a rupture test at 1,500° F and the specimen is briefly heated to higher temperatures, cooled back to 1,500° F, and restressed there is reduction in rupture life at 1,500° F in comparison with that obtained in the usual test at constant load and constant temperature. The degree to which rupture life was reduced was a function of the conditions under which the overheating occurred:

(1) For specimens which were overheated periodically during the entire test (fig. 6), the degree of reduction of time for rupture increased as the overheat temperature was increased. Overheating to 1,650° F had little effect on the rupture time, while overheating to 1,800° and 1,900° F caused significant reductions. Overheating to 2,000° F resulted in the most severe damage, the rupture times being reduced to 288 from 1,210 hours for a stress of 16,200 psi and to 25 from 94 hours for a stress of 22,000 psi. The data suggest that the overheating may also increase the slope of the curves of stress against rupture time somewhat.

One way to appreciate the magnitude of the effect of overheating is to consider the change in rupture strengths indicated by the cyclically overheated tests of figure 6. Continuous cycling to failure using the two schedules at the 22,000- and 16,200-psi stresses gives the following values:

Overheat temperature, °F	Stress for rupture, psi, at 1,500° F in -	
	100 hr	1,000 hr
None	21,500	16,500
1,650	21,500	16,000
1,800	20,800	14,800
1,900	20,000	13,800
2,000	18,300	13,800

These figures represent the maximum effects observed. Lesser amounts of overheating reduced the strength less. Consideration of these rupture strengths is perhaps more realistic than consideration of rupture times which are subject to much wider variations. It should also be recognized that the frequency of overheating could influence the results and the above values are for the specific conditions used in the tests.

(2) For any one temperature of overheating, the percent of damage for a given amount of overheating appeared to be greater for tests run at 16,200 psi than for those run at 22,000 psi (fig. 7). While the difference in cycle frequency may have been a factor, the data obtained were not sufficiently extensive to substantiate it.

(3) At each overheat temperature, the degree of reduction in life increased as the overheat cycling was continued for an increasing percentage of the total life at 1,500° F (fig. 8). The intermediate points represent tests in which overheating was stopped at the indicated accumulated overheat time and the test allowed to continue to rupture at constant load and temperature without interruption. The test conditions for this generality should be kept in mind since the schedule of overheats in relation to the progress of the rupture test may influence the results. Therefore, it should be clearly recognized that the curves of figure 8 on which this generality is based are for tests in which the 5- or 12-hour cycles of overheat were applied from the start of the tests. Also, as figure 3 shows, the accumulated time at overheat temperature also includes any effects due to being exposed to temperatures above 1,500° F during heating and cooling.

(4) Figure 8 also indicates that there was definite tendency, particularly for the tests run with a stress of 16,200 psi (rupture time of 1,210 hours at 1,500° F), for a saturation point for no further damage from additional overheating to exist. That is, there is for each temperature some amount of accumulated overheat time which caused maximum reduction of rupture time and overheating more than this amount did not result in any further damage. The one exception to this generality was tests overheated to 1,900° F, where no saturation effect was observed at either stress.

(5) The loss in life for the first few overheats increased markedly (fig. 8) as the overheat temperature was increased to 1,900° and 2,000° F. A limited number of overheats at 1,650° and 1,800° F may have increased life slightly and certainly was not significantly harmful. Two overheats of 2-minute duration affected the rupture time at 1,500° F for 16,200-psi stress as follows:

Overheat temperature, °F	Rupture time at 1,500° F, hr
None	[a]1,210
1,650	1,330
1,800	1,210
1,900	850
2,000	360

[a]Normal rupture test.

(6) The effect of overheating at various points in the rupture life at 1,500° F is not well established by the data. Overheating for a total of 5 cycles of 2 minutes to 1,800° F with the first cycle delayed until 160 hours had elapsed at 1,500° F under 16,200 psi gave slightly greater damage than is indicated by the curve for tests overheated at the start (figs. 7(b) and 8). The data are somewhat incomplete at short overheat times to 1,800° F and this may not be truly significant. Delaying the start of overheating to 1,900° F for the same amount of time apparently resulted in the opposite effect. That is, the test lasted longer than material overheated the same amount from the start (figs. 7(c) and 8). This again was not significantly far from the curve drawn through the data for overheating continuously from the beginning of the test.

(7) The data obtained on material preheated to the overheat temperature and then rupture-tested in a normal fashion are presented in figures 9 and 10 together with some data on work of this type from other laboratories. In order to consider all of these data on a common basis, calculations were made of the percent of normal rupture time obtained for each of the preheat conditions. All of the data then could be plotted on a comparative basis. This may be somewhat questionable, however, since the curves have been drawn for percentage of life changes regardless of testing stresses which varied from 16,200 psi for the University of Michigan tests to the 25,000 psi used by General Electric Co. and Allegheny Ludlum Steel Corp. It should also be noted that the curve for 1,650° F preheating shown in figure 10 represents an interpolation in figure 9 between data at 1,600° and 1,800° F, since only limited data were available at this temperature. These two figures indicate the following:

(a) Overheating prior to testing showed an increase in rupture life when the overheat temperature was 1,600° or 1,650° F. This suggests that the increase in life previously indicated for a limited number of overheats to 1,650° F during rupture-testing was real and not data scatter. It will be noted that the preheat tests at 1,600° F show that the amount of increase in life from heating to 1,600° F diminished with increased time of heating. Furthermore, comparison of the curves for overheats to 1,650° F in figures 8 and 10 shows remarkable agreement, indicating that for overheating to this temperature the effect is the same whether applied before testing or during the test.

(b) For temperatures of 1,800° F and higher, there is no question that preheating does not give the same effect as overheating during the test. Comparison of figures 8 and 10 shows that cycling to these temperatures during the test results in more severe damage. The only possible exception to this would be short times at 1,800° F.

(8) In all of the above tests the load was cycled as well as the temperature, introducing the possibility that load cycling alone appreciably influenced results. To investigate this, a test was run at each of the two stresses during which the load was cycled with the same frequency as in the overheat tests, but the temperature was kept constant at 1,500° F. The results of these tests are given in table II and plotted in figure 8. Apparently life was reduced under the lower stress and increased under the higher stress. Both points lie just outside the range of values for ordinary rupture tests. It seems best to conclude that load cycling does have an effect but that it is relatively small in comparison with overheat effects when the overheat temperatures are 1,800° to 2,000° F.

The difference in response to overheating at the two stresses may be partly the result of this load-cycling phenomenon. The tests at 22,000 psi showed less damage percentagewise for a given amount of overheating than those at 16,200 psi. By comparison, the test with load cycling at 22,000 showed improvement in strength while that at 16,200 showed damage. The two effects seemed to be just large enough to suggest that both were significant. Calculations of the percentage loss in life from overheating were carried out using the rupture times indicated by the cyclic-load tests instead of the nominal rupture times in constant-load tests. This did not bring the results any closer together for tests at 16,200- and 22,000-psi stress. Consequently, it was concluded that load-cycling effects were not responsible for the lack of agreement on a percentage basis for temperature damage at the two stress levels.

<u>Effect on elongation</u>.- Measurements of total elongation at fracture are included in the respective tables for each type of overheating. Figures 11(a) and 11(b) show these data for tests at 16,200 and 22,000 psi, respectively. The following generalities are indicated by figure 11(a) for tests under 16,200 psi:

(1) The tests of specimens cyclically overheated to temperatures from 1,650° to 2,000° F indicated the following trends:

(a) Overheating limited times to 1,800°, 1,900°, and 2,000° F reduced elongation in the rupture tests at 1,500° F. The effect increased with overheat temperature. Apparently, overheating to 1,650° F had little effect.

(b) Increasing the number of overheats at the higher temperatures resulted in an increase in elongation back toward the nominal steady-temperature rupture-test values. It almost appears as if saturation in rupture-time damage results in normal ductility.

(c) Delayed overheats and simple load cycling at 1,500° F also reduced ductility but in a somewhat erratic manner.

(2) Preheating before testing did not reduce ductility as much as did cyclic overheating and, in some cases, increased the ductility over that of a normal test. The tests at 1,600° F for the shorter time periods did indicate a possible slight reduction.

The data for tests under 22,000 psi (fig. 11(b)), although rather sparse, indicate that, with the possible exception of overheating to 1,650° F, overheating resulted in a decrease in ductility for all the temperatures considered. This decrease was greater the longer the time of overheating. Therefore, the effects of overheating on ductility at 1,500° F seem to vary depending on the stress level at 1,500° F or possibly the frequency of overheating.

<u>Effect on creep curves of overheating in absence of stress</u>.- Creep data were taken for all tests. The time-elongation plots of these data are presented in figures 12 and 13. In figure 12 every point plotted for the overheat tests represents an overheat. In figure 13 the points indicating overheats are plotted only during the limited periods that overheating was involved in the test. Points noted as standard creep readings are routine measurements taken after overheating had been discontinued or before overheating was begun. Also included in these figures is the curve for no overheats and that for overheating to failure. From consideration of these figures the following generalities can be made:

(1) For cyclic overheating continuously to failure from the beginning of the test (fig. 12), creep was accelerated with the degree of acceleration increasing as the overheat temperature increased from 1,650° to 2,000° F. This generality was influenced by the 1,500° F stress level in the following way:

(a) At a stress of 16,200 psi, overheating apparently increased creep from the start of the tests (fig. 12(a)).

(b) When the stress at 1,500° F was 22,000 psi, it appeared as if little effect occurred until considerable overheating had accumulated (fig. 12(b)), except when the overheat temperature was 2,000° F. At this temperature, the creep curve showed faster creep from the first overheat.

(2) The effects of limited amounts of overheating (fig. 13) are considerably more difficult to generalize. This arises from the complexity of the situation as well as from the scarcity of data. The following statements are made on the basis of tests overheated from the start of the tests:

(a) Stopping overheats before rupture never resulted in an increase in rate of creep over that of continuing overheats to fracture.

(b) If the accumulated overheat time before stopping the overheating was sufficient for maximum damage to rupture strength, the rate of creep afterwards was essentially the same as if overheats were continued to rupture.

(c) If the accumulated overheat time was not sufficient to reach a maximum reduction in rupture strength, creep rates decreased after the overheats below those characteristic of continued overheating. The rates, however, always were higher than for tests with no overheats at an equivalent total deformation. From this it appears that limiting overheats according to the schedule of the experiments can be expected to increase creep after overheat in proportion to the degree of damage from overheating.

(3) Delaying the start of overheating as well as limiting the number of overheats introduces a far more complex situation. This could be expected to lead to the following sequence of events:

(a) Creep will proceed to the point of overheating as indicated by normal constant-temperature tests.

(b) During the overheating, creep accelerates to approximately the same rate as in continuously overheated tests at the same total deformation. This, however, is based on only two tests, both at the 16,200-psi stress level (figs. 13(c) and 13(e)).

(c) The creep rates remained higher after stopping the overheats than for the tests without overheats but were lower than for continuously overheated material. As far as could be judged, the effects were similar in magnitude to an equivalent amount of overheat from the start of the tests.

The test with one overheat of 5-minute duration at 1,800° F after prior creep for 315 hours (fig. 13(c)) fairly well agrees with the above generalization. The exact magnitude of the effect appears, however, to have been exaggerated by an abnormally weak specimen for the test, as can be seen by comparing the creep curve prior to overheating with that for the normal rupture test.

(4) Heating before starting the tests had the same types of effects as were noted in the tests for rupture times. Figure 14 shows these data and indicates the following results:

(a) Heating at 1,600° F increased creep resistance except for the longest time of preheating.

(b) Heating at 1,800° and 2,000° F reduced creep resistance but not as much as an equivalent amount of cyclic heating during the tests.

(5) Cycling the load at 1,500° F did not significantly alter the creep at 22,000 psi (fig. 12(b)). At 16,200 psi, however, there appeared to be a slight but significant increase in creep rate as a result of load cycling (fig. 12(a)).

Effect on time to reach a given total deformation of overheating in absence of stress.- Figures 15 and 16 present various plots of times to reach given values of total deformation taken from the creep curves of figures 12(a), 13(a), 13(c), 13(e), and 13(g):

(1) Figure 15 shows the time to reach a given amount of total deformation as a function of the overheat temperature for tests which were cycled in the absence of stress every 12 hours until failure occurred at 1,500° F and 16,200 psi. The creep curves from which these points were taken are in figure 12(a). The following points may be noted from figure 15:

(a) As the overheat temperature increased, the time to reach any of the indicated deformations became less. This decrease in time was quite uniform on the logarithmic time scale. That is, the curves for different amounts of deformation remained roughly parallel over the entire temperature range.

(b) In terms of actual time increments, this reduction in time represents a rather severe effect. For example, the time to reach 1-percent strain in the normal rupture test was 40 hours. For the test overheated to 2,000° F, this time was 12 hours, a decrease of 60 percent. Proportional reductions can be observed for lower overheat temperatures and different values of deformation.

(2) Figure 16 shows the effect of increasing amounts of overheating on the time required to reach a given total deformation. These curves show for limited overheating from the start of the test that as the number of overheats is increased the time to reach a given deformation is reduced. The maximum reduction in this time, under the fixed overheating schedule employed, was reached when overheating was continued until the total deformation of interest was reached. As a result of this fixed schedule, the maximum change in the time required to reach a given deformation was fixed by the number of overheats that was possible before this deformation was attained. As the amount of total deformation considered increased, there was time for more overheats and, therefore, opportunity for a more severe decrease in the time required to reach this deformation. The straight line sketched in on each plot at which the curves terminate is thus merely a plot of the maximum number of overheats which could be accumulated at any time under the schedule which was used.

The following additional points should be noted regarding the construction of these figures:

(a) Data for these figures were taken from the creep curves of figures 13(a), 13(c), 13(e), and 13(g) and thus are specifically indicative only of results obtained using the cycles employed for these tests, that is, one overheat every 12 hours from the start of the test in the absence of stress with the stress at 1,500° F being 16,200 psi.

(b) For many conditions data were not available. In these cases, the best curve possible was sketched through the existing points, taking account of its relation to the other existing curves around it.

Overheating in Presence of Stress

The purpose of the portion of the overall program devoted to overheating in the presence of stress was to determine the way in which the effect of stress combines with the temperature effects as discussed in the preceding section to produce a given final test result. The data for these tests are given in table IV. Creep curves for the tests are shown in figures 17 and 18.

The amount of testing where overheats were conducted in the presence of stress was rather limited. The general approach was to determine if the calculated amount of rupture life used up by creep plus the loss by temperature damage would account for the observed rupture times. Proof or disproof of this possibility by a few tests was thought to be the best way to develop general principles from the relatively few tests possible within the limitations of the program.

Principle of calculation.- In analyzing the data obtained from these tests, the following general formula was postulated and applied:

$$t_o = t_n - \left(d_t + d_s\right)$$

where

t_o time for rupture in overheat test

t_n normal time for rupture under stress used at 1,500° F

d_t reduction in rupture time resulting from temperature damage during overheat

d_s reduction in rupture time resulting from presence of stress at overheat temperature

These factors were evaluated as follows:

(1) The life lost due to temperature cycling d_t was estimated from the measured effects of cyclic overheating in the absence of stress accumulated in the previous section. Because the overheats under stress were conducted at 1,500° F under a stress of 18,000 psi (average rupture time, 500 hours), it was necessary to estimate the damage effect from the available data on tests conducted at 22,000 and 16,200 psi. This was done by averaging the percentage reduction in life by cycling at both stresses.

(2) The percent of life used up under stress at the overheat temperature d_s was calculated by dividing the total time under stress at the overheat temperature by the normal rupture time under the stress at the overheat temperature. The total time under stress was obtained by summing the number of 2-minute overheats applied.

Results for overheating to 1,650° F.- One test was conducted with overheats to 1,650° F holding the stress constant at 18,000 psi at both 1,500° and 1,650° F. The rupture time was 348 hours and the total number of overheats was 27. The damage was computed as follows:

(1) Computation of temperature damage: Consideration of the curves of loss in rupture life against accumulated time of overheating to 1,650° F (fig. 8) indicates that no significant loss would be expected from 54 minutes at 1,650° F.

(2) Computation of stress damage:

(a) One test at 1,650° F and 18,000 psi gave a normal rupture time of 852 minutes (14.2 hours).

(b) There were 27 2-minute overheats before rupture which is a total time of 54 minutes at 1,650° F under 18,000 psi.

(c) 54/852 = 6.4 percent of the total rupture life used up at 1,650° F by creep.

(3) The ranges in rupture times at 1,500° F under 18,000 psi indicated by figure 5 for normal rupture tests, less a loss by stress damage of 6.4 percent for the time at 1,650° F, were as follows:

	Average	Minimum	Maximum
Normal rupture time, hr	495	360	570
Reduced by 6.4 percent, hr	463	337	534
Rupture time of overheat test, hr			348

The creep curves (fig. 17) show how the short overheats at 1,650° F add substantial amounts of creep and cause the creep curve to deviate from the constant-temperature creep curve at 1,500° F.

Results for overheating to 1,800° F.- Three tests were conducted with overheats to 1,800° F with stress present.. In one test the stress was left at 18,000 psi and one overheat applied. The rupture time was 347 hours. The computed damage was as follows:

(1) Temperature damage: Two minutes at 1,800° F apparently had no significant effect on rupture life as judged from figure 8.

(2) Computation of stress damage:

(a) Four rupture tests at 18,000 psi and 1,800° F gave rupture times ranging from 10 to 23 minutes. The two extremes were tests heated by resistance as in the overheat tests. Two tests brought to a uniform temperature of 1,800° F in a furnace and then loaded as in a normal rupture test both had rupture times of 18 minutes (table I and fig. 4).

This indicates that the usual stress-rupture test at 1,800° F does not give values seriously different from the conditions of the overheat test and that the rupture time of 18 minutes could be used for computing stress damage.

(b) Stress damage for one overheat of 2 minutes is 2/18 = 11.1 percent of the total available rupture life used up at 1,800° F.

(3) Computation of expected life:

	Average	Minimum	Maximum
Normal rupture time at 1,500° F, hr	495	360	570
Reduced by 11.1 percent, hr	440	319	506
Rupture time of overheat test, hr			347

Two tests, in which the stress was reduced to 12,500 psi during the overheat cycles to 1,800° F, gave rupture times of 156 and 187 hours. The computation of the damages yields as follows:

(1) Computation of temperature damage: One of the two tests lasted for 14 cycles, the other, 13. This gives an average time at 1,800° F of 27 minutes. Consideration of figure 8 indicates a life left after 27 minutes of heating of approximately 60 percent of the normal life. This indicates that the damage due to temperature cycling was about 40 percent of the available life.

(2) Computation of stress damage: Testing in a normal rupture test at 12,500 psi and 1,800° F gave rupture times ranging from 2.7 to 3.3 hours which averaged to 3.05 hours (see table I). This indicates a damage from 27 minutes of 27/60/3.05 or about 15 percent of the available rupture life.

(3) Computation of expected life: The total damage as calculated then is 40 + 15 percent or 55 percent, which should result in a rupture time 45 percent of normal. The values obtained were:

	Average	Minimum	Maximum
Normal rupture life at 1,500° F, hr	495	360	570
Reduced by 55 percent, hr	223	162	256
Actual rupture time, hr			187 and 156

The creep curves for all three of these tests overheated to 1,800° F under load appear in figure 18. The tests run with the stress reduced during the overheat show curves in good agreement with each other. The creep rates between overheats are accelerated over that obtained for a constant-temperature test, as would be expected from the combined effects of the temperature and stress on the creep properties. The test given a single overheat to 1,800° F with full load also shows evidence that subsequent creep was accelerated by the stress damage during the overheat.

Summarized results of overheats under stress.- The actual values for rupture with overheats under stress are all within the probable range of computed values although on the low side in every case. This could be a matter of chance or it could indicate that the actual damage is more than was indicated by the two principles used in computing the rupture times. It seems highly probable that computing rupture life by these principles gives quite good but somewhat high results.

Probably the most important point to recognize is that substantial loss in life can occur with relatively small amounts of overheat when:

(1) A stress which is relatively high in relation to the rupture strength at the overheat temperature is present during overheating.

(2) The stress is relatively low but the overheat temperature is as high as 1,900° to 2,000° F where temperature alone can introduce large amounts of damage.

Microstructural Effects

Metallographic examinations of specimens after rupture indicated that overheating had two general visible effects:

(1) Overheating to 1,650° and 1,800° F probably accelerated precipitation and agglomeration of the phase or phases which normally form during testing at 1,500° F. (See figs. 19, 20(a), and 20(b).) Apparently continuous cycling every 12 hours to 1,650° F had little effect on final structure while that at 1,800° F broke up the network of precipitates in the grain boundaries.

(2) Overheating to 1,900° F continuously to rupture reduced the general precipitate and slightly agglomerated the grain-boundary precipitates (fig. 20(c)). Raising the temperature to 2,000° F nearly completely eliminated both general and grain-boundary precipitation (fig. 20(d)).

Figures 21(a) and 21(b) show that a few overheats to 1,900° F gave intermediate amounts of restriction of general precipitation. In addition, a very fine general background precipitate was present. Figures 21(c) and 21(d) show more or less the same thing where the tests were stopped at the end of the overheats and not carried to rupture. As would be expected, there was not so much precipitation in these samples, particularly when overheating was stopped after five cycles, because of the reduction of time at 1,500° F where the major part of the precipitation occurs. The fine background precipitates present in the samples continued to rupture (figs. 21(a) and 21(b)) were absent.

Figures 21(e), 21(f), and 21(g) show microstructures of samples given limited amounts of overheating to 2,000° F. From these can be seen the restriction and coarsening of general precipitates from 1 cycle of 2 minutes at 2,000° F and a pronounced reduction in general precipitation from 2 cycles and 10 cycles. In these specimens, it must be recognized that there was opportunity for precipitation to take place at 1,500° F after temperature cycling was stopped. Figure 22 shows the complete absence of general precipitation when a sample was fractured at 2,000° F in about an hour in a rupture test.

Careful examination of the microstructure of samples overheated to 1,900° and 2,000° F, particularly at 100 diameters, suggests increases in size of the columbium carbonitride particles as the amount of general precipitate decreases.

The General Electric Co. had circulated a chart of photomicrographs entitled "Overtemperature Study of S-816 Alloy" showing "solution effects" in S-816 alloy as a function of temperature and time. This has been used by several organizations to estimate temperatures of overheating. The General Electric chart was based on reheating material simply given the standard solution treatment and age at 1,400° F. The tests of this investigation also had varying lengths of time at 1,500° F under stress and most of the samples had ruptured at 1,500° F.

The curves from the General Electric chart have been reproduced in figure 23 and points from the present investigation have been superimposed. It will be noted that there is indication that more or less the same microstructural changes resulted from periodic overheating during rupture tests at 1,500° F as are shown by the General Electric chart for simply reheating after aging. It was quite surprising that approximately the same total time was required for noticeable and reasonably complete disappearance of excess phases under the two conditions. The precipitation and agglomeration during testing at 1,500° F apparently did not alter the total time-temperature relationship for a given structure change. In making this comparison the accumulated total time for the 2-minute overheats was used.

Mechanism of Damage from Overheating

Overheating can change the strength of S-816 alloy by structural alteration and by using up available creep-rupture life through temperature-acclerated creep if stress is present during the overheat. Apparently, damage due to stress being present can be approximated quite closely by computing the percentage of the normal constant-temperature rupture time under stress at the overheat temperature. The mechanism for the loss in life due to structural changes induced by temperatures alone is less clear.

The microstructure exhibited by the various specimens suggests that exposure to temperatures in the range of 1,650° to 1,800° F results first in accelerated precipitation at 1,500° F followed by agglomeration and overaging. At 1,900° to 2,000° F, it almost appears as if solution of the excess phases occurs. Solution, however, is not by any means clearly established as the basic mechanism.

One or two exposures of 2-minute duration to 2,000° F reduced rupture life by a very large amount. It would seem that the reaction rate was rather rapid to be explained by solution of excess phases. Furthermore, experience with alloys of the type of the S-816 alloy indicates that solution of excess phases does not reduce and usually increases rupture life. If simple solution were the answer, it would seem that overheating should not be so damaging.

The very rapid rate of damage from overheating at 2,000° F suggests that some critical precipitate dispersion was broken up. There is, furthermore, the inference that this is probably submicroscopic in nature. Most experience indicates that when precipitate particles become as large as those visible in S-816 alloy they are incoherent and too widely dispersed to be very effective in retarding creep. It is believed that a good deal of the strength of S-816 alloy at 1,500° F is dependent on some submicroscopic coherent-type precipitate mechanism involving minor alloying elements such as carbon or nitrogen. Actually, there was metallographic evidence of a fine precipitate in those samples overheated to a limited extent to 1,900° F. It seems highly probable that this precipitate represents agglomeration and growth of the critically dispersed coherent precipitates which are the major factor in variation of creep-rupture strength of S-816 alloy under the conditions of this investigation.

There seemed to be considerable microscopic evidence, under conditions of the cyclic overheats during tests at 1,500° F, that solution of excess phases was not occurring. Instead, it appeared as if the columbium carbonitride particles were increasing in size. It is believed that this probably was the major reason for the disappearance of excess phases from repeated overheats to 2,000° F. It seems quite possible that carbon and/or nitrogen could react with columbium and precipitate out under the overheat conditions. This would remove the carbon and/or nitrogen from solution and they would not be available to form the usual carbides while at 1,500° F.

The damage from heating the alloy at 1,900° to 2,000° F seems to be sensitive to the prior history of the alloy. Heating at these temperatures before testing is not as damaging as overheating during exposure to stress at 1,500° F. There must be some reaction involved in the prior exposure to 1,500° F or to creep at 1,500° F. In this connection, other investigators of overheating have informally reported that overheat damage at these temperatures with even a small amount of stress present cannot be recovered by subsequent heat treatment at 2,150° F. This could be conveniently explained by the transfer of carbon and/or nitrogen to inactive columbium carbonitrides, as discussed in the preceding paragraph. Such compounds would be expected to resist re-solution and prevent rejuvenation of high-temperature strength by heat treatment. The difficult part to explain is the absence of any permanent damage from preheating to these temperatures before testing. There are a number of reasons to question simple solution as the cause of overheat damage.

While the data are not conclusive and a good deal of speculation is involved, it seems probable that the temperature damage from overheating involved in structural changes can be summarized as follows:

(1) Overheating to 1,600° to 1,650° F for short time periods increases creep resistance by acceleration of a submicroscopic coherent precipitate reaction involving carbon and/or nitrogen. Longer overheating to these temperatures apparently causes slight overaging and slight loss of strength. There seems to be a definite end point beyond which further overheating has no effect.

(2) Increasing the overheat temperature to 1,800° F increases the rate of overaging and the extent to which it progresses. This reaches a definite end point for a stress of 16,200 psi at 1,500° F, for the overheat cycles used, beyond which further overheating has no effect.

At the same time, there is an increase in the rate of formation and agglomeration of the microscopically visible precipitates which form at 1,500° F. It is doubtful that an increase in the size of particles already visible microscopically has much effect on creep-rupture strength.

(3) Increasing the overheat temperature to 1,900° F increases the rate of overaging of the submicroscopic aging reaction. Visible particles develop as a result of limited overheating to this temperature. The end point of the reaction was not reached under the conditions of cyclic overheating used in this investigation. It was also apparently accelerated

and extended by either exposure to 1,500° F and for creep at this temperature or cyclic overheats of short duration during creep-rupture tests at 1,500° F. Such cyclic overheats caused more damage than simple heating at 1,900° F prior to testing at 1,500° F.

At the same time, carbon and/or nitrogen starts to precipitate out on existing columbium carbonitrides. This begins to deplete the available carbon and/or nitrogen for precipitate formation while the material is at 1,500° F under stress.

(4) Increasing the overheat temperature to 2,000° F very rapidly destroys the submicroscopic aging reaction. Only very brief overheating is required for this to occur when overheats occur during rupture tests at 1,500° F.

The transfer of carbon and/or nitrogen to existing columbium carbonitrides is accelerated at 2,000° F. The time at 2,000° F for cycles of 2-minute duration twice a day was almost sufficient to transfer all the carbon and/or nitrogen to this inactive form and prevent precipitation at 1,500° F. This also appears to be the ultimate end of the elements vital to the submicroscopic aging reaction.

(5) The main effect of cyclic overheating during a test at 1,500° F seems to be to carry the damaging reactions further than simple overheating before testing. The visible microstructural changes seem to be about the same in both cases.

The data were carefully reviewed for evidence that recovery from strain-hardening arising from creep was a major factor in the temperature-actuated overheat damage. It would be convenient to explain the results in terms of this phenomenon. This would get around the difficulty of unequal effects from overheating prior to and during tests at 1,500° F for the same gross microstructural changes. However, for recovery to have been a major factor, it would be difficult to account for the saturation points beyond which further overheating caused no more damage at 1,650°, 1,800°, and 2,000° F, but not at 1,900° F. Recovery would be expected to operate as long as overheats were applied. To account for observed effects, only structural changes and not recovery would be important for temperatures up to 1,900° F. At 1,900° F, recovery would have to be slow and therefore effective throughout the tests. When the temperature was raised to 2,000° F, recovery would have to be quite rapid and only effective for primary creep to account for the rapid saturation effect. The case for recovery being anywhere near as important as alteration of a precipitate type of reaction seems doubtful.

Interpretation of Results in Terms of Overheating in Gas Turbines

The testing program indicated that overheating to 1,650°, 1,800°, 1,900°, and 2,000° F reduces creep-rupture life of S-816 alloy. The degree of damage increases with temperature and time at temperature. Damage arises from structural changes induced in the alloy by temperature effects and, if a stress is present during overheating, by an accelerated loss of life by creep at the higher temperature.

In a gas turbine overheating could occur at any time in the creep-rupture life of the metal. Presumably, the number of overheats would also be very limited in number. The following reasoning then could be used to analyze the probable effect of any specific case:

(1) An overheat early in the life of the turbine could be evaluated from the data presented in this report.

(2) If an appreciable percentage of the life had been used up by normal service prior to overheating, the data are less certain. Certainly only the subsequent service life would be affected. The data from this investigation cannot define the effects completely. Apparently, however, the damage is about the same anywhere in a test. Consequently, the remaining life would be reduced and the overall life reduced proportionally less. For S-816 alloy at 1,500° F, this is somewhat academic because the creep extension would be so large by the time the rupture life had been half used up that few applications could stand this much deformation. Consequently, the data presented should cover the majority of applications.

(3) Limited overheats of 2-minute duration early in creep-rupture life of S-816 alloy would reduce life at 1,500° F by temperature damage as follows:

Overheat temperature, °F	Rupture time, hr, at 1,500° F under stress normally causing rupture in indicated time periods	
	100 hr	1,000 hr
	One 2-min overheat	
1,650	98	1,050
1,800	95	1,030
1,900	95	900
2,000	85	350
	Two 2-min overheats	
1,650	98	1,100
1,800	90	1,000
1,900	90	700
2,000	75	300
	Five 2-min overheats	
1,650	95	1,200
1,800	80	700
1,900	80	550
2,000	30	250

It will be noted that one or two overheats have relatively little effect except at 2,000° F. Also, the percentage loss increases with the nominal rupture time.

In estimating damage from actual overheats, it could be expected that:

(1) The percentage damage would increase with the increase in the nominal rupture times. Thus, if the actual operating stress allowed a normal rupture time of several thousand hours, then the reduction in life would be more than the percentage indicated by the above figures.

(2) The longer the service before overheating occurred, the less the total service life would be affected because only the remaining life would be changed. Possibly the remaining life would be reduced by about the same percentage as is indicated by the data in this report for overheating early in the rupture life. The little data available on delayed overheats tend to support this possibility.

It would seem that the most serious problem from a relatively few overheats insofar as creep-rupture life is concerned would be a rather high temperature or the presence of a relatively high stress during an overheat.

Review of the data suggests that insofar as S-816 alloy at 1,500° F is concerned, the probability is that a few short-duration overheats would not in most cases drastically reduce rupture strength. Thus, it is probable that in many cases other effects of overheating, such as thermal shock damage, will be far more important than the effects on creep-rupture properties.

CONCLUSIONS

The following conclusions were drawn from an investigation of overheating S-816 alloy to temperatures of 1,650°, 1,800°, 1,900°, and 2,000° F during the course of rupture tests at 1,500° F:

1. Overheating at temperatures up to 2,000° F reduced rupture life of S-816 alloy at 1,500° F. The loss in life increased with both temperature and accumulated time of overheating. The only exception was some increase in rupture life from limited overheating to 1,600° and 1,650° F in the absence of stress.

2. The loss in rupture strength arises from both alteration of the alloy structure by temperature effects and, if stress is present, by the temperature acceleration of creep. The presence of appreciable stress during overheating would be the predominant source of damage. Even very brief exposure at 1,800° to 2,000° F under the stresses normally causing rupture in 100 to 1,000 hours at 1,500° F would either exhaust a large proportion of the rupture life or cause immediate rupture.

3. Temperature alone can reduce rupture times at 1,500° F to a pronounced extent. Two overheats to 2,000° F of 2-minute duration with stress removed reduced the rupture time at 1,500° F under 16,200 psi from an average of 1,210 hours to 349 hours. The effects were less at lower temperatures with the reduction from overheating at 1,650° F hardly being significant for even a large number of such overheats.

4. The combined effects of temperature and creep damage for overheating in the presence of stress can be computed reasonably well. The loss in life from temperature cycling must be added to the loss in life by creep. Estimation of the temperature damage requires prior measurement of the effect of overheat temperature on rupture time at 1,500° F.

The creep damage can be estimated as the percent of total available rupture time at the overheat temperature represented by the actual time at the overheat temperature.

5. Temperature alone induces internal structure changes which influence strength at 1,500° F. Two overheats of 2-minute duration affected the rupture time at 1,500° F for 16,200 psi as follows:

Overheat temperature, °F	Rupture time at 1,500° F, hr
None	[a]1,210
1,650	1,330
1,800	1,210
1,900	850
2,000	360

[a]Normal rupture test.

Thus, one or two overheats become significant only when the temperature is 1,900° F or higher.

6. Cyclic overheating, with stress removed during the overheats, until fracture occurred at 1,500° F disclosed that a saturation in damage occurred for temperatures of 1,650°, 1,800°, and 2,000° F. After a specific number of overheats no further damage occurred. These saturation times were accumulated during tests under 16,200 psi (normal rupture time, 1,210 hours) at 1,500° F but not for tests under 22,000 psi (normal rupture time, 94 hours). When overheated at 1,900° F, saturation was not attained.

7. Microstructural studies indicated that overheating to 1,650° and 1,800° F accelerated the normal precipitation and agglomeration process which occurs during testing at 1,500° F. Apparently heating to 1,900° and 2,000° F caused some part of those alloying elements which participate in normal precipitate formation during testing at 1,500° F, probably carbon and/or nitrogen, to be transferred to inactive columbium carbonitrides. This reduces precipitate formation during tests at 1,500° F and, when the total time at 2,000° F is sufficient to carry the reaction to completion, gives the appearance of a solution-treated structure. This, however, apparently does not account for the very pronounced loss in strength from very brief overheating at 2,000° F. The main loss in rupture life occurred before the overheat time was extensive enough to cause a recognizable change in the microscopically visible structure of

the alloy. This suggests that there is probably a submicroscopic coherent precipitate containing carbon and/or nitrogen which provides high strength and that this is destroyed by very limited heating to 2,000° F.

8. Repeated cyclic overheats cause the temperature damage to be more extensive and to occur faster than heating the test material to the same temperatures before testing. Consequently, overheating before testing cannot be used to predict temperature damage reliably in S-816 alloy.

9. A limited number of overheats at any time during the creep-rupture life apparently has about the same effect as if they were applied early in the test. Because such overheats can affect only the future life after the overheat, the overall loss in life diminishes as overheating is delayed toward the end of the rupture test.

University of Michigan,
Ann Arbor, Mich., March 9, 1956.

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TABLE I

RUPTURE TESTS ON S-816 ALLOY

Temp., °F	Stress, psi	Rupture time, hr	Elongation, percent	Reduction of area, percent
1,500	15,400	1,628	40	47
	16,200	1,110	43	52
		1,242	46	47
		1,215	50	49
		1,268	47	30
	16,400	804	45	49
	17,500	507	46	49
	18,000	435	39	23
		600	45	26
	18,500	364	38	24
	19,400	237	44	54
	19,600	206	60	55
	21,600	95	61	50
	22,000	106	50	43
		82	40	41
	22,600	65	62	50
1,650	18,000	14.2	37	16
	25,000	1.7	33	17
1,800	11,500	4.8	17	23
	12,500	2.7	27	23
		3.0	26	12
		3.1	26	13
		3.3	25	9
	16,200	.6	17	22
		.7	26	28
	18,000	.30	27	2
		.30	35	13
		[a].17	18	15
		[a].38	17	15
1,900	16,200	.07	26	21
2,000	6,000	.92	24	11
	4,000	4.8	19	6

[a]Tests run to rupture maintaining temperature by resistance heating.

TABLE II

CYCLIC OVERHEATS IN ABSENCE OF STRESS ON S-816 ALLOY

[All cycles 2 min. except those noted]

Overheat temp., °F	No. of cycles	Rupture time, hr	Fraction of rupture life, percent	Elongation, percent	Reduction of area, percent
1,210-hr rupture stress of 16,200 psi					
1,500	[a]69	828.9	68.5	34	44
1,650	[b]76	894.2	73.8	47	31
	8	1,541.9	127.3	48	48
1,800	[b]44	524.5	43.5	47	36
	25	467.1	38.6	44	26
	[c]5	677.7	56.0	35	32
	[d]1	798.0	66.1	54	(e)
	2	1,261.0	104.2	36	24
1,900	[b]31	368.5	30.4	36	46
	15	511.2	42.2	39	40
	[c]5	774.5	64.0	40	24
	3	677.2	56.0	34	24
	1	1,051.8	87.0	32	26
2,000	[b]24	287.8	23.7	42	26
	10	291.0	24.1	30	15
	5	315.6	26.1	29	17
	2	349.1	28.9	30	17
	1	438.6	36.2	28	20
94-hr rupture stress of 22,000 psi					
1,500	[a]15	127.9	136.0	48	50
1,650	[b]16	84.3	89.7	46	23
	10	84.0	89.6	42	20
	5	90.0	95.7	35	20
1,800	[b]13	66.3	70.5	32	19
	8	69.7	74.2	43	23
1,900	[b]11	55.8	59.4	33	20
	6	66.4	70.6	(e)	20
2,000	[b]5	24.9	26.5	18	6
	3	58.6	62.3	33	16

[a]Specimen with load cycled at 1,500° F until failure.
[b]Overheated to failure.
[c]First overheat delayed until 160 hrs.
[d]5-min cycle delayed 410 hrs.
[e]Specimen damaged when removed from holders.

TABLE III

RUPTURE TESTS ON PREHEATED BARS OF S-816 ALLOY

Preheat temp., °F	Preheat duration, min	Rupture time		Elongation, percent	Reduction of area, percent	Normal rupture test (a)
		hr	percent of normal			
1,600	30	2,137	175	45	43	1
	60	1,586	130	42	41	1
	240	1,070	89	57	49	1
	240	49	91	25	40	3
1,650	130	65	93	31	(c)	2
	240	48	89	(b)	(b)	3
1,800	5	990	82	47	45	1
	10	68	97	33	(c)	2
	20	1,056	87	42	39	1
	240	45	83	31	41	3
1,950	7	34	49	31	(c)	2
	240	37	69	(b)	(b)	3
2,000	10	923	77	55	43	1
	30	801	67	40	23	1
	240	35	65	30	46	3

[a]Normal rupture tests:

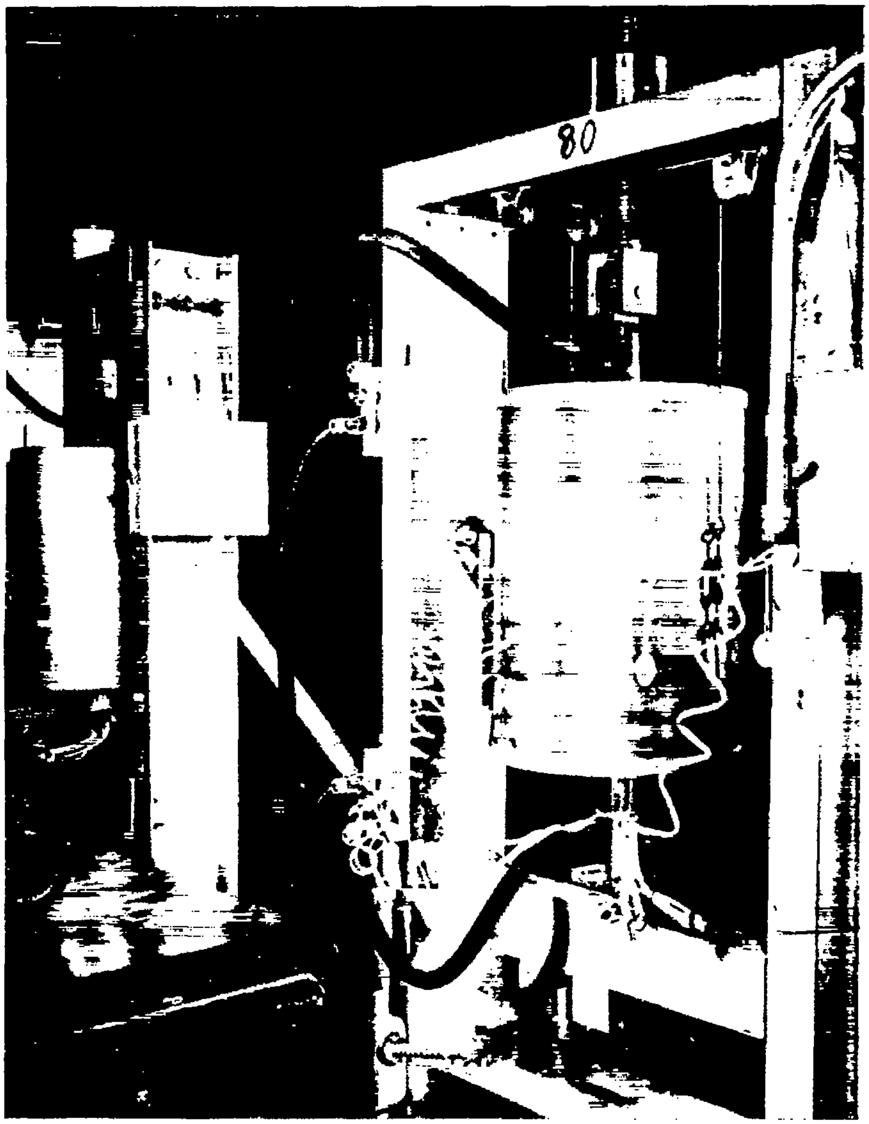
Designation	Tests from -	Stress, psi	Time, hr	Elongation, percent	Reduction of area, percent
1	Present investigation	16,200	1,210	48	45
2	General Electric Co. (ref. 3)	25,000	65	28	(c)
3	Allegheny Ludlum Steel Corp. (ref. 4)	25,000	54	30	34

[b]Rupture times interpolated from plot.
[c]Reduction of area not reported.

TABLE IV

OVERHEATS IN PRESENCE OF STRESS ON S-816 ALLOY

Normal stress, psi	Overheat stress, psi	Overheat temp., °F	No. of cycles	Rupture time, hr	Elongation, percent	Reduction of area, percent	Expected life, hr
18,000	12,500	1,800	14	187	38	22	223
	12,500	1,800	13	156	37	24	223
18,000	18,000	1,800	1	347	44	23	440
18,000	18,000	1,650	27	348	39	46	463



L-57-4145

Figure 1.- Photograph showing creep-rupture unit modified for use in overheating by resistance heating.

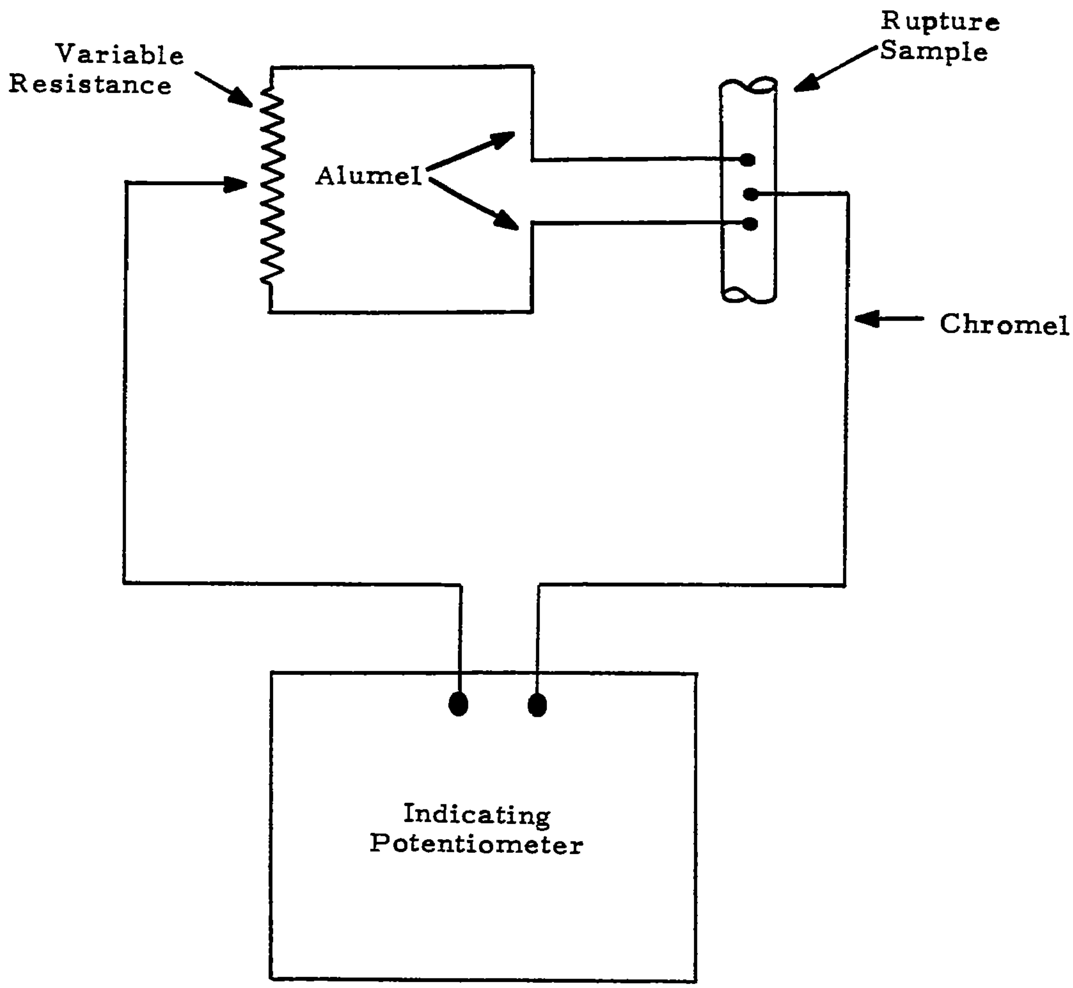


Figure 2.- Schematic wiring diagram of system used for measurement of temperature during overheats to avoid extraneous electromotive force from heating current.

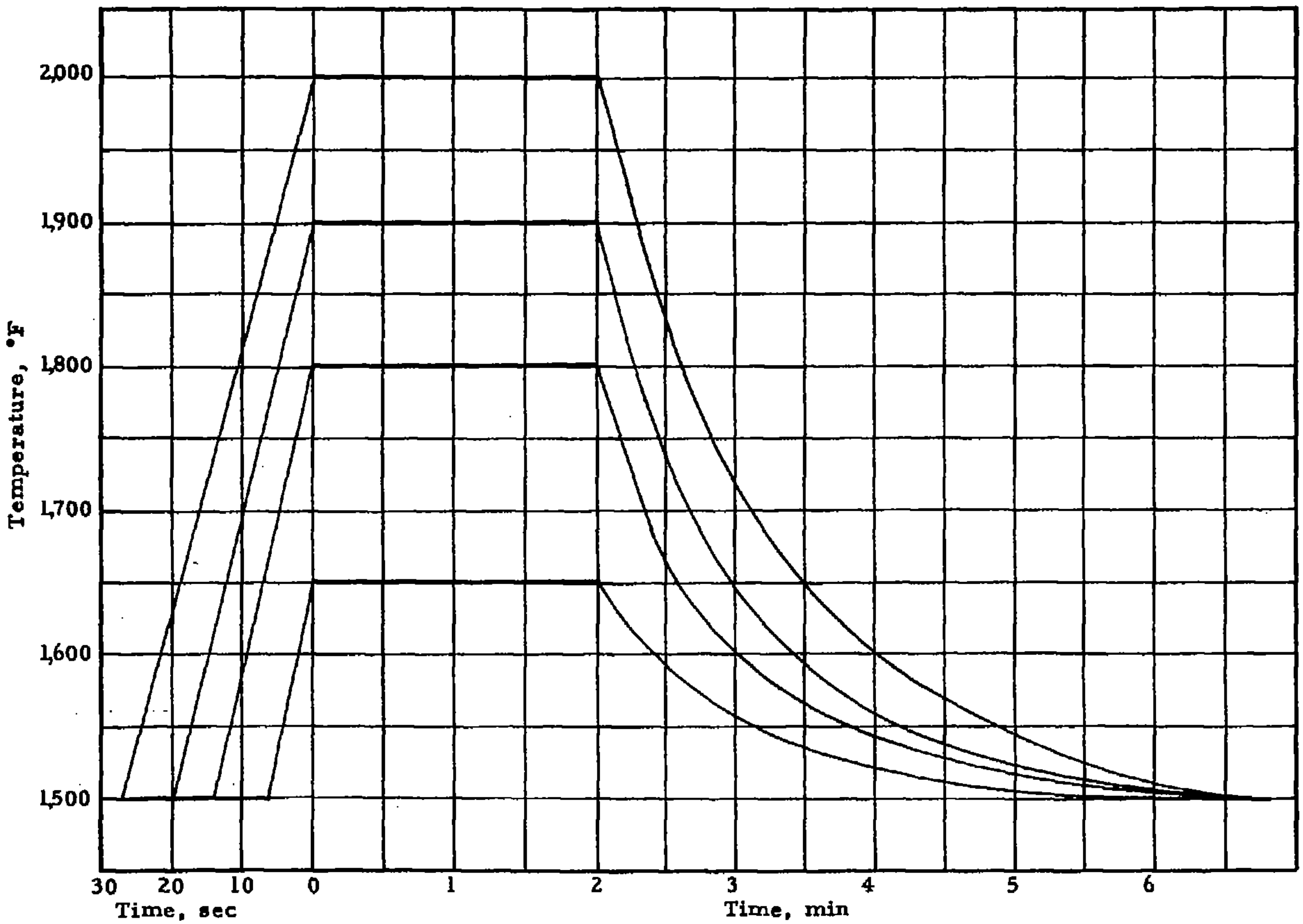


Figure 3.- Typical time-temperature curves for overheats to each of temperatures employed.

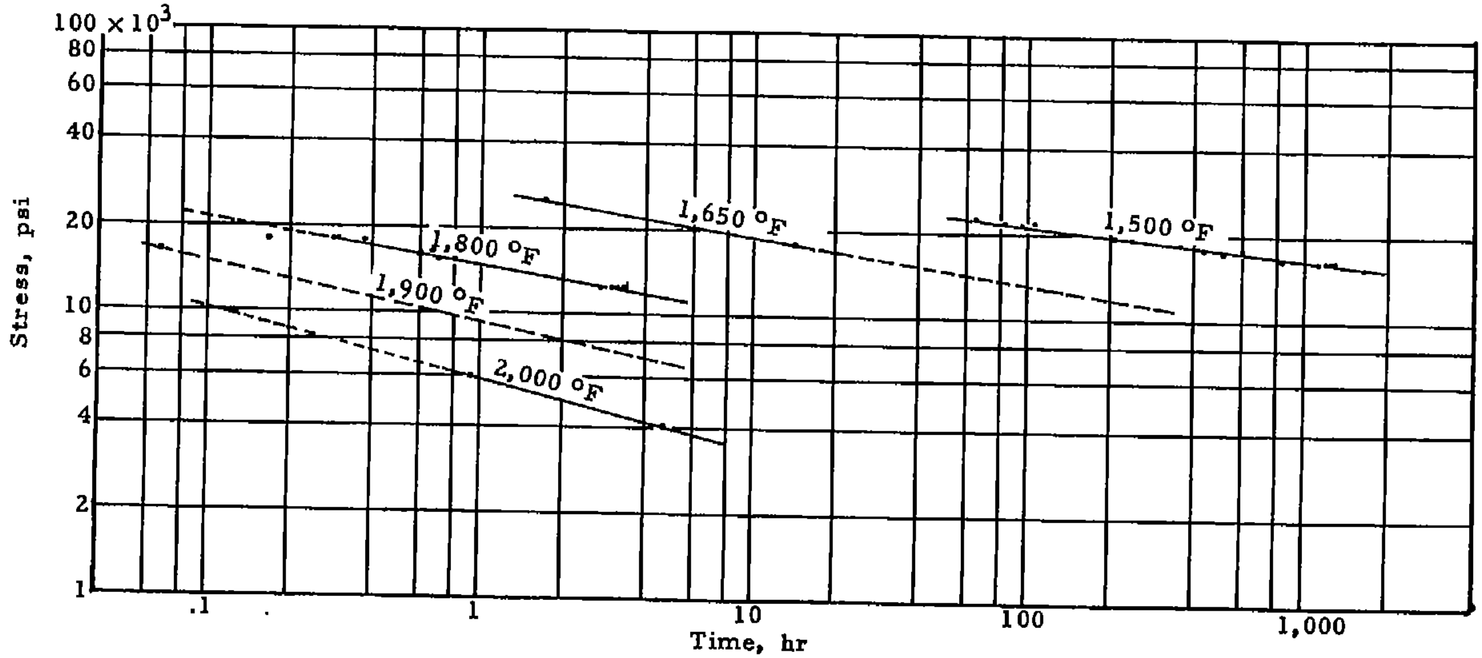


Figure 4.- Normal constant-temperature stress-rupture data at 1,500°, 1,650°, 1,800°, 1,900°, and 2,000° F for S-816 experimental material.

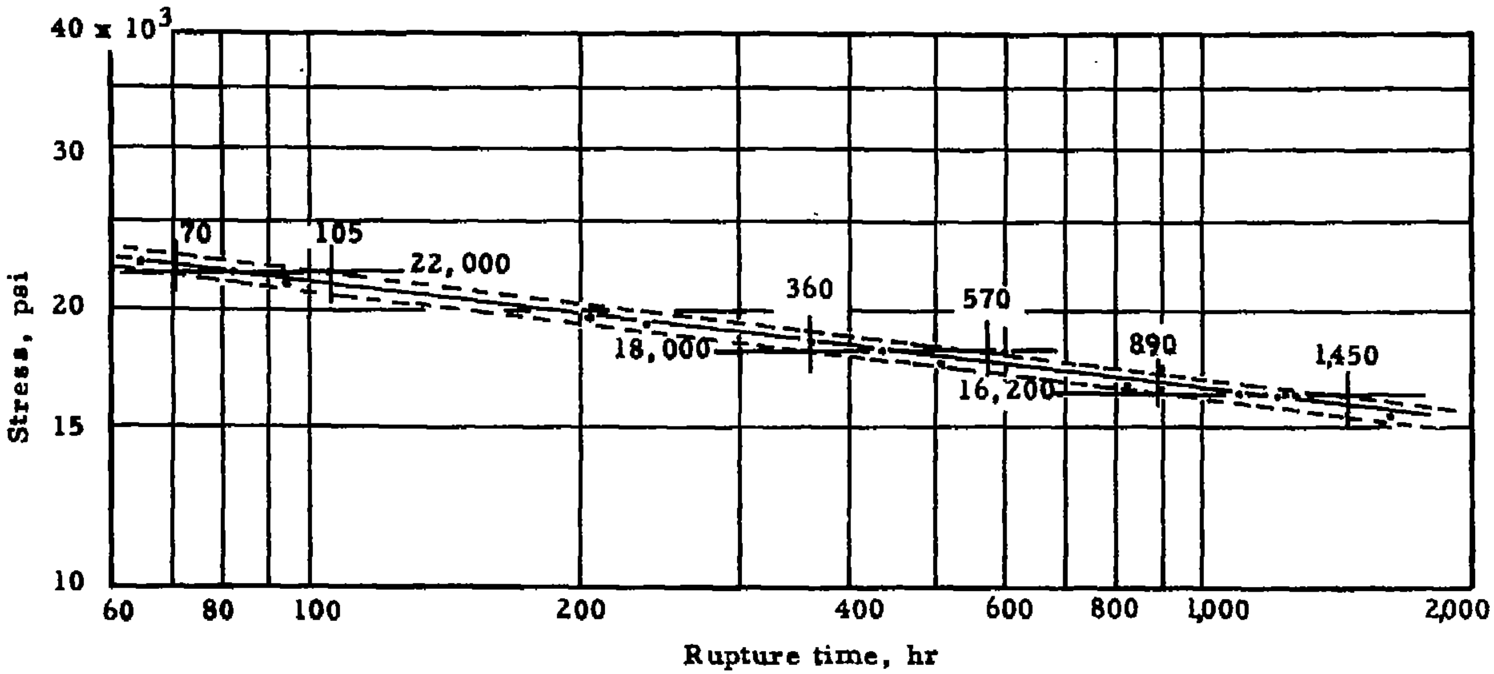


Figure 5.- Curve of stress against rupture time at 1,500° F for S-816 alloy showing ranges in rupture times predicted by available test data for three stresses used in this investigation.

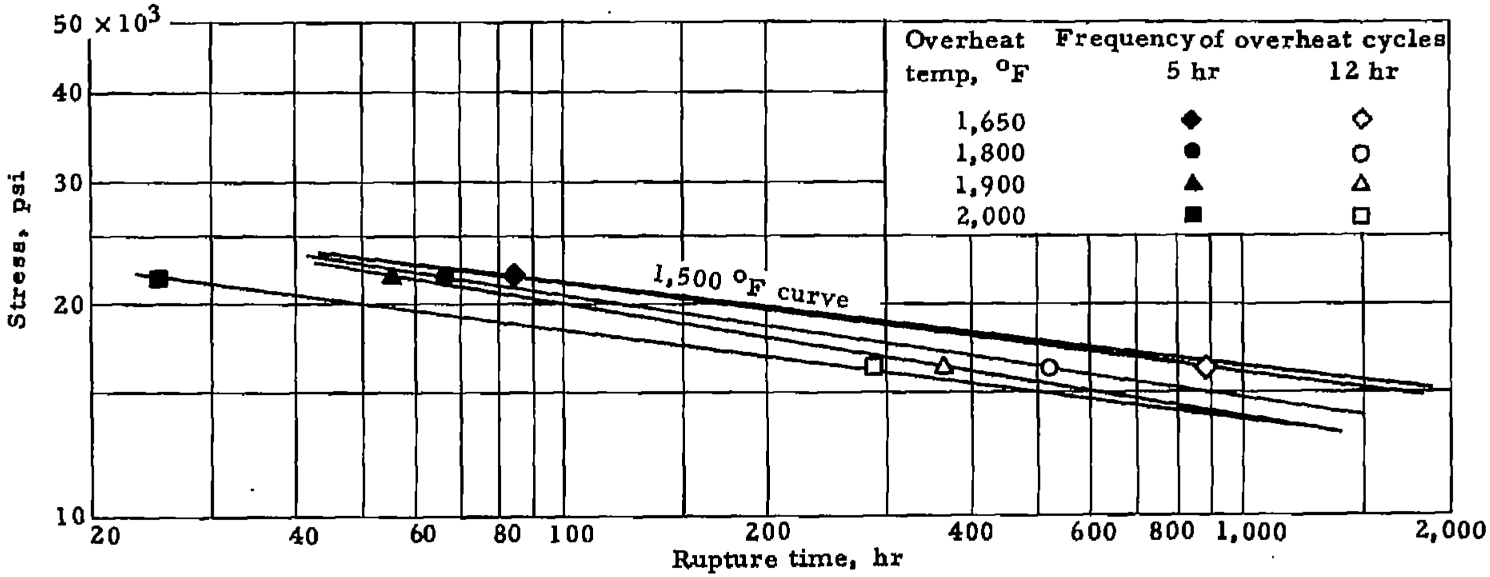
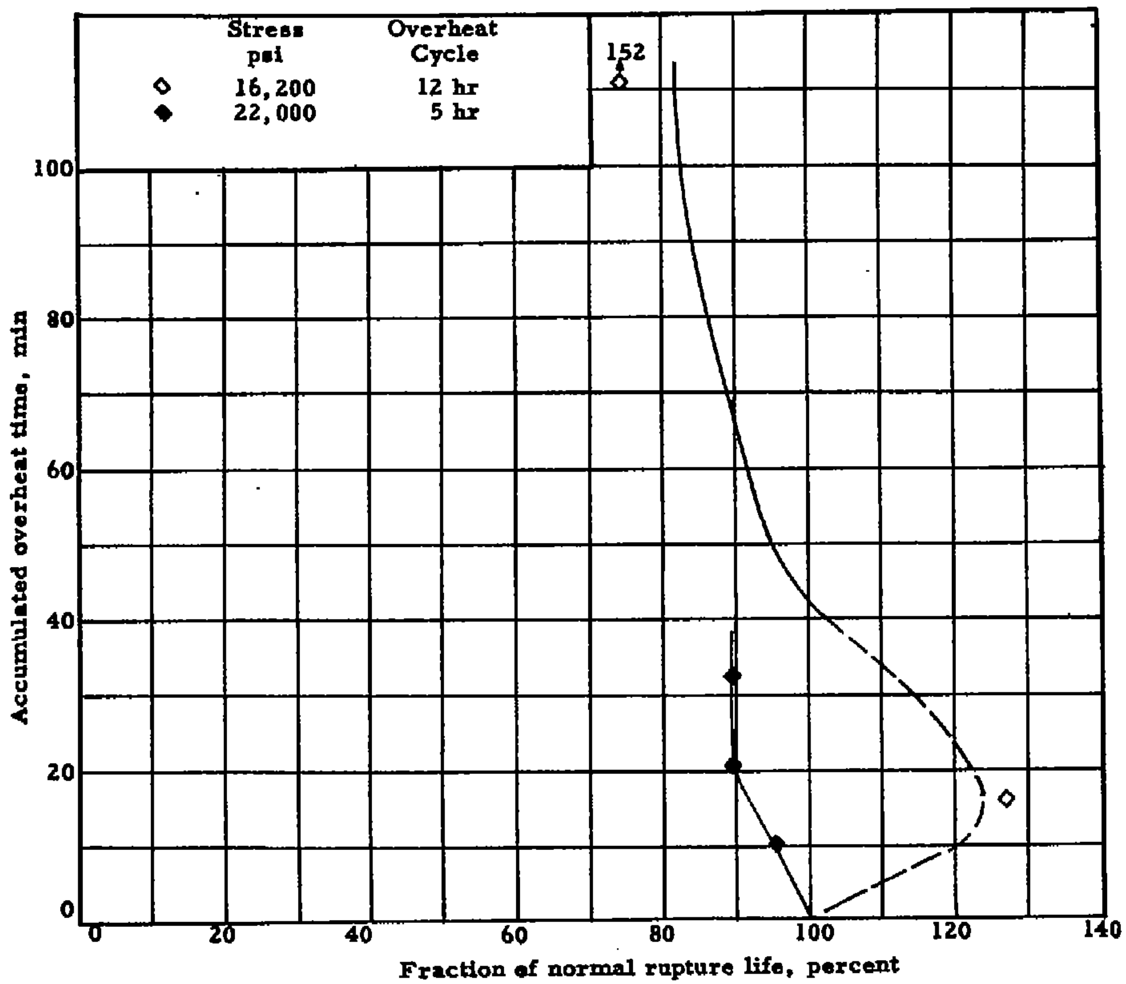
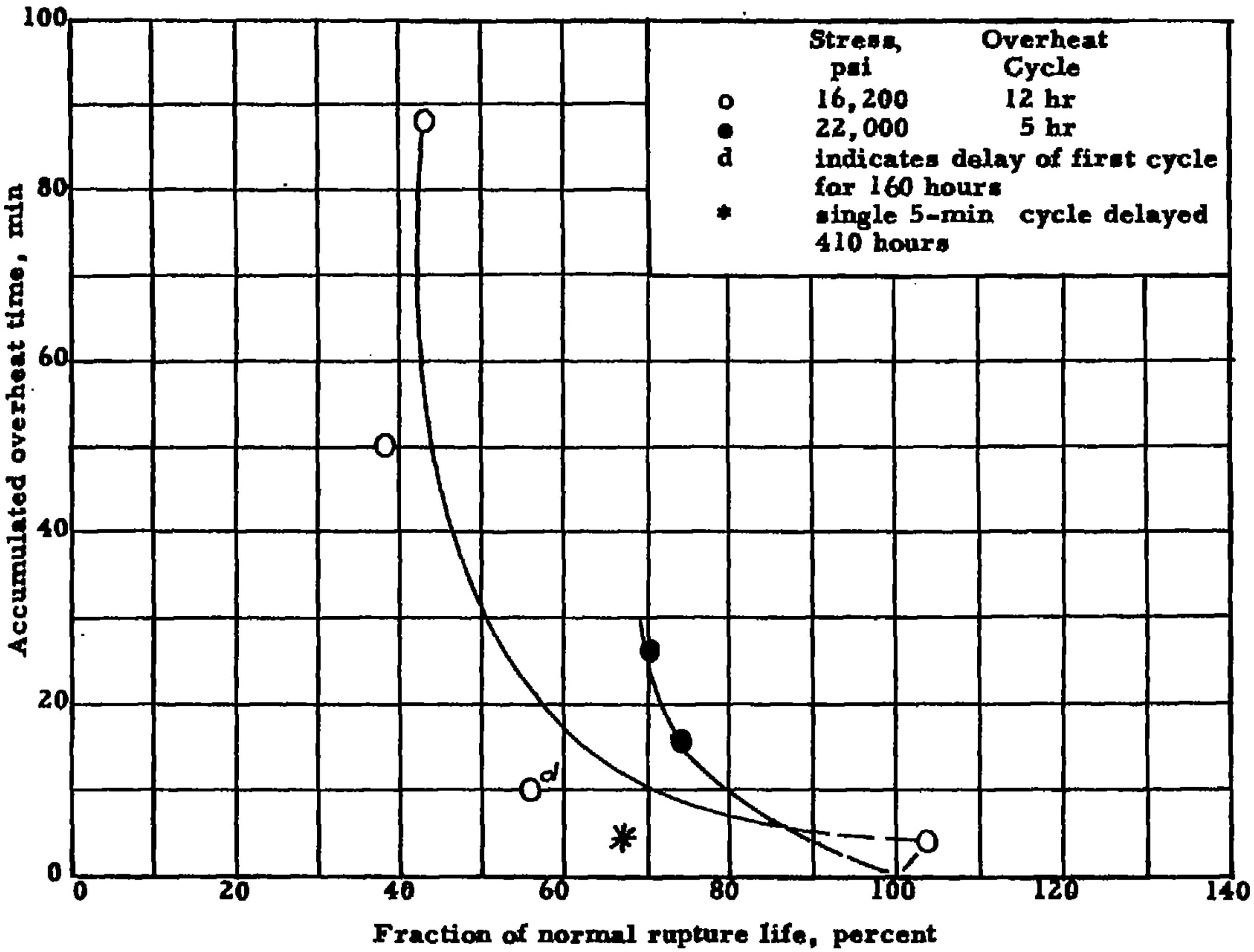


Figure 6.- Influence of continued cyclic overheating to 1,650°, 1,800°, 1,900°, and 2,000° F in absence of stress on rupture time at 1,500° F. Stress was removed during each 2-minute overheat.



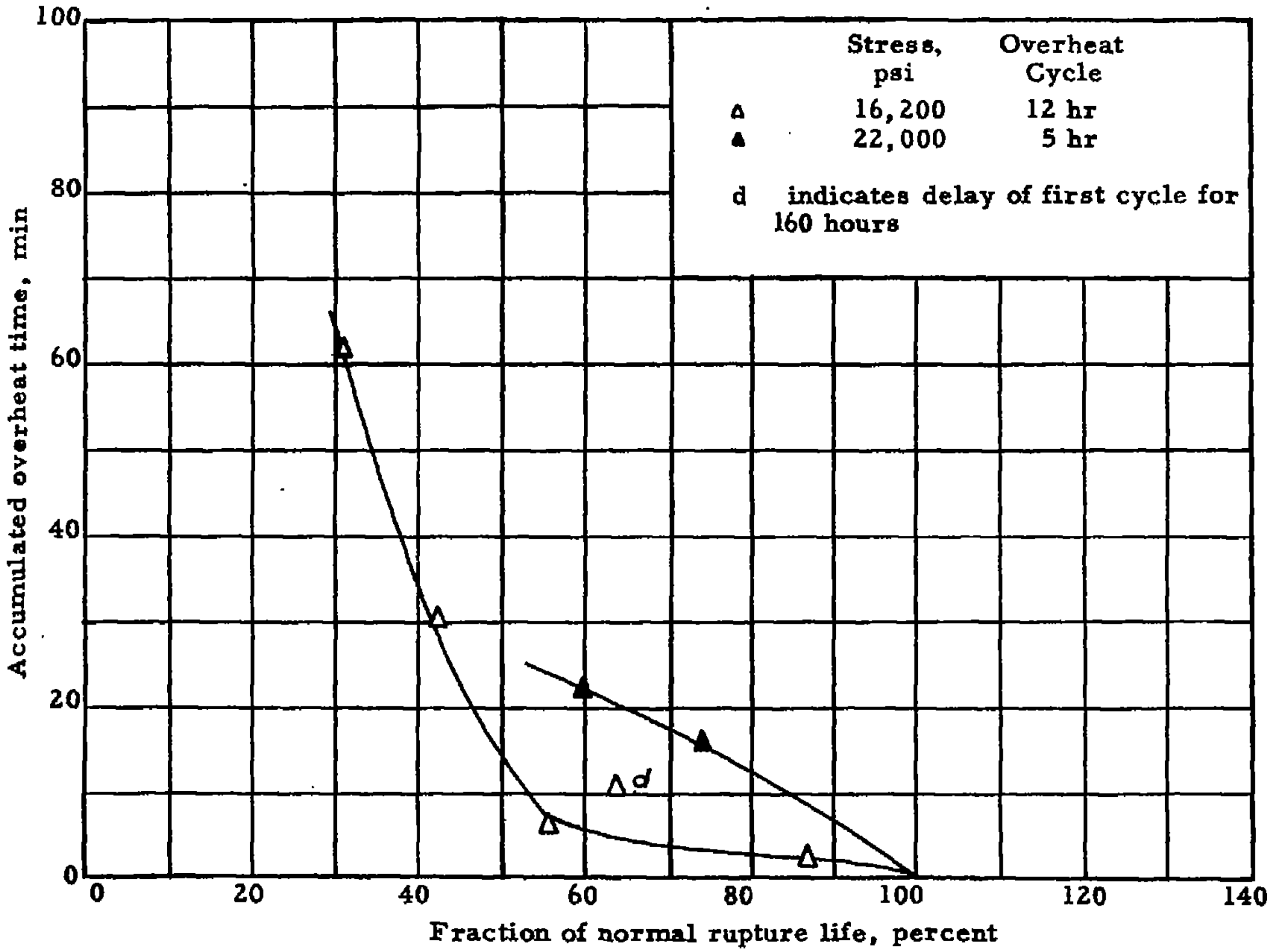
(a) Overheating to 1,650° F.

Figure 7.- Effect of amount of overheating to various temperatures on rupture life at 1,500° F under stresses of 22,000 and 16,200 psi. Stress removed during 2-minute overheats applied every 5 and 12 hours.



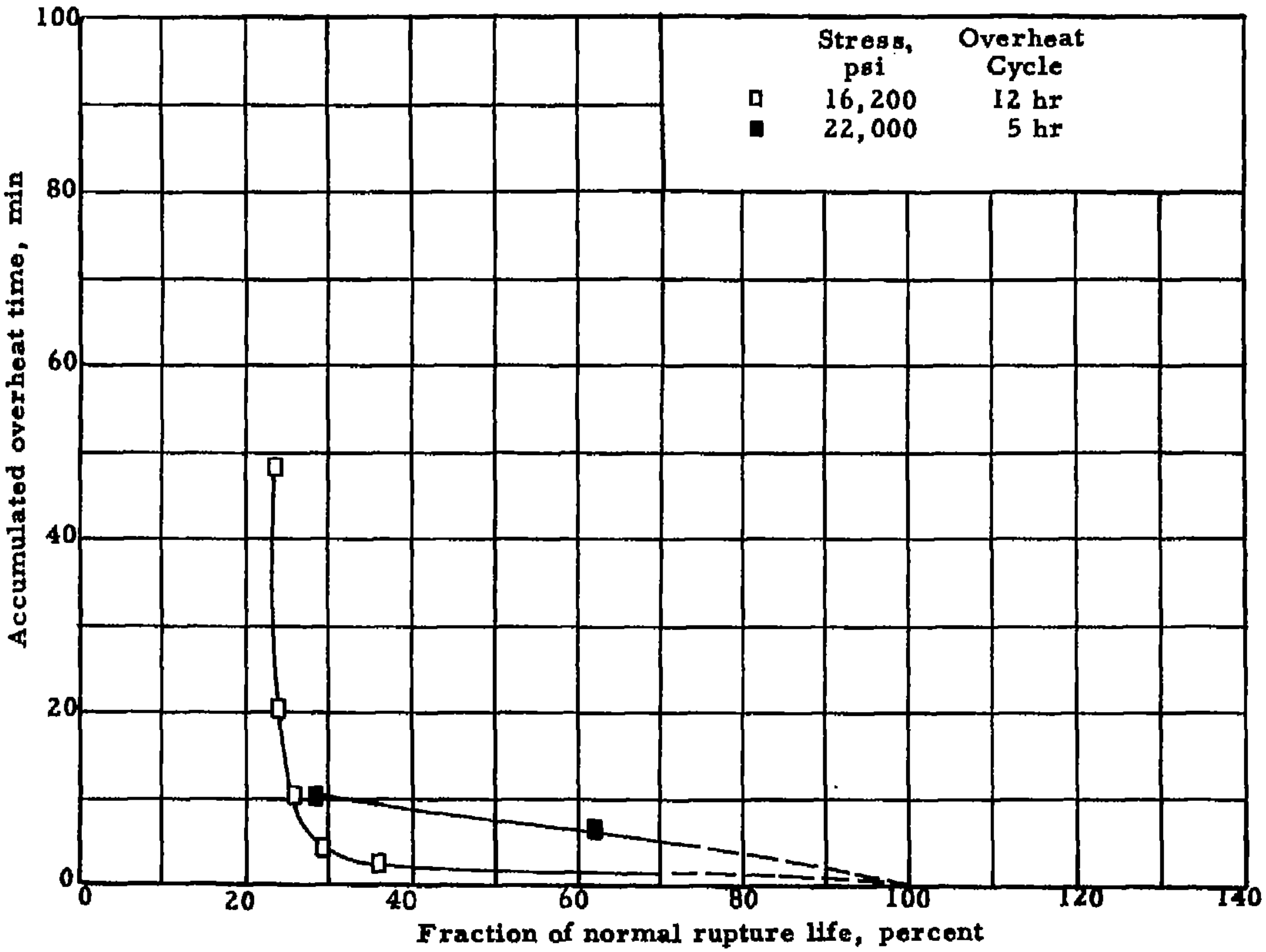
(b) Overheating to 1,800° F.

Figure 7.- Continued.



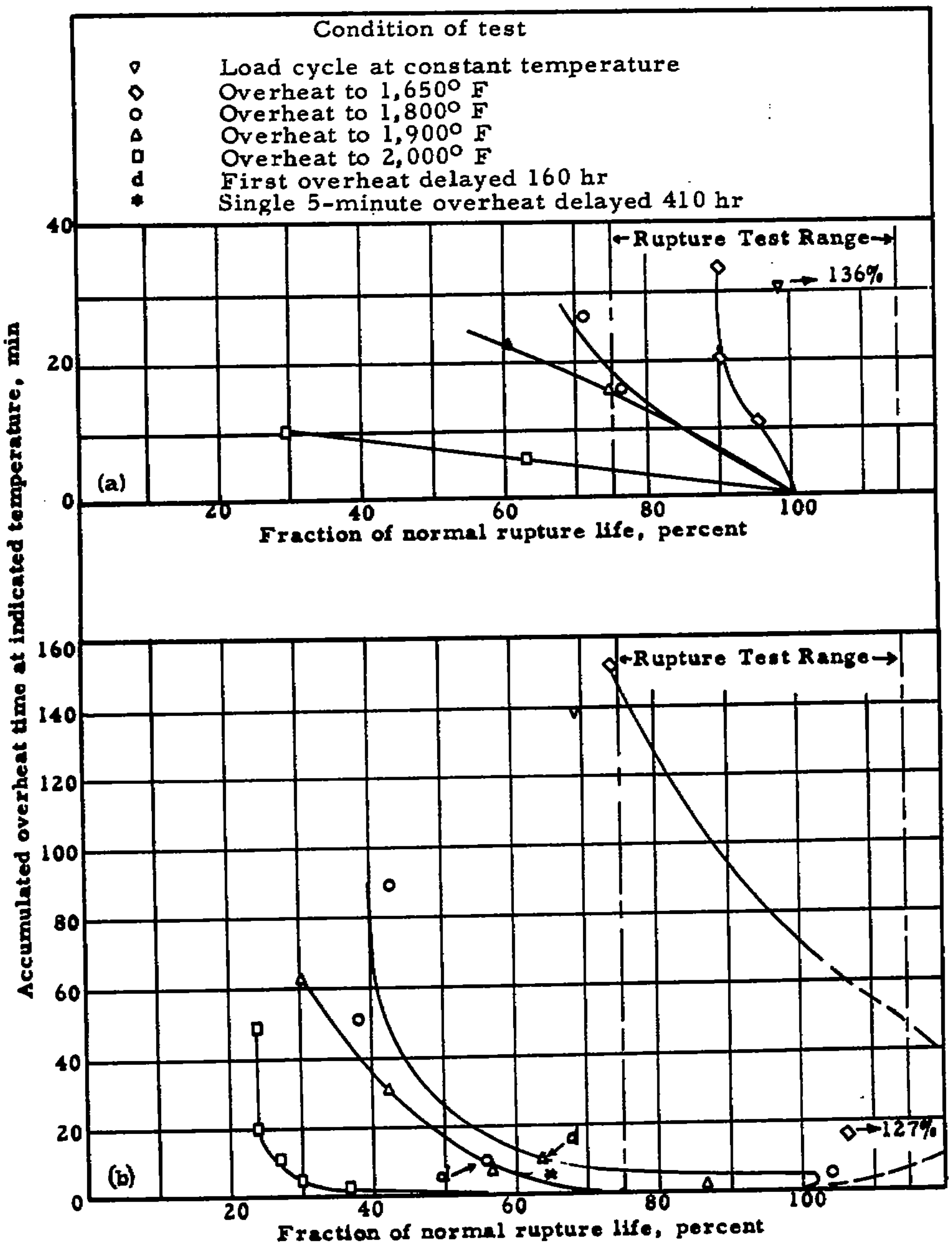
(c) Overheating to 1,900° F.

Figure 7.- Continued.



(d) Overheating to 2,000° F.

Figure 7.- Concluded.



(a) Tests at 1,500° F and 22,000 psi (normal rupture time, 94 hours) using 2-minute overheat cycles every 5 hours.

(b) Tests at 1,500° F and 16,200 psi (normal rupture time, 1,210 hours) using 2-minute overheat cycles every 12 hours.

Figure 8.- Effect of amount of overheating to 1,650°, 1,800°, 1,900°, and 2,000° F on rupture life at 1,500° F under stresses of 16,200 and 22,000 psi. Stress removed during overheat period.

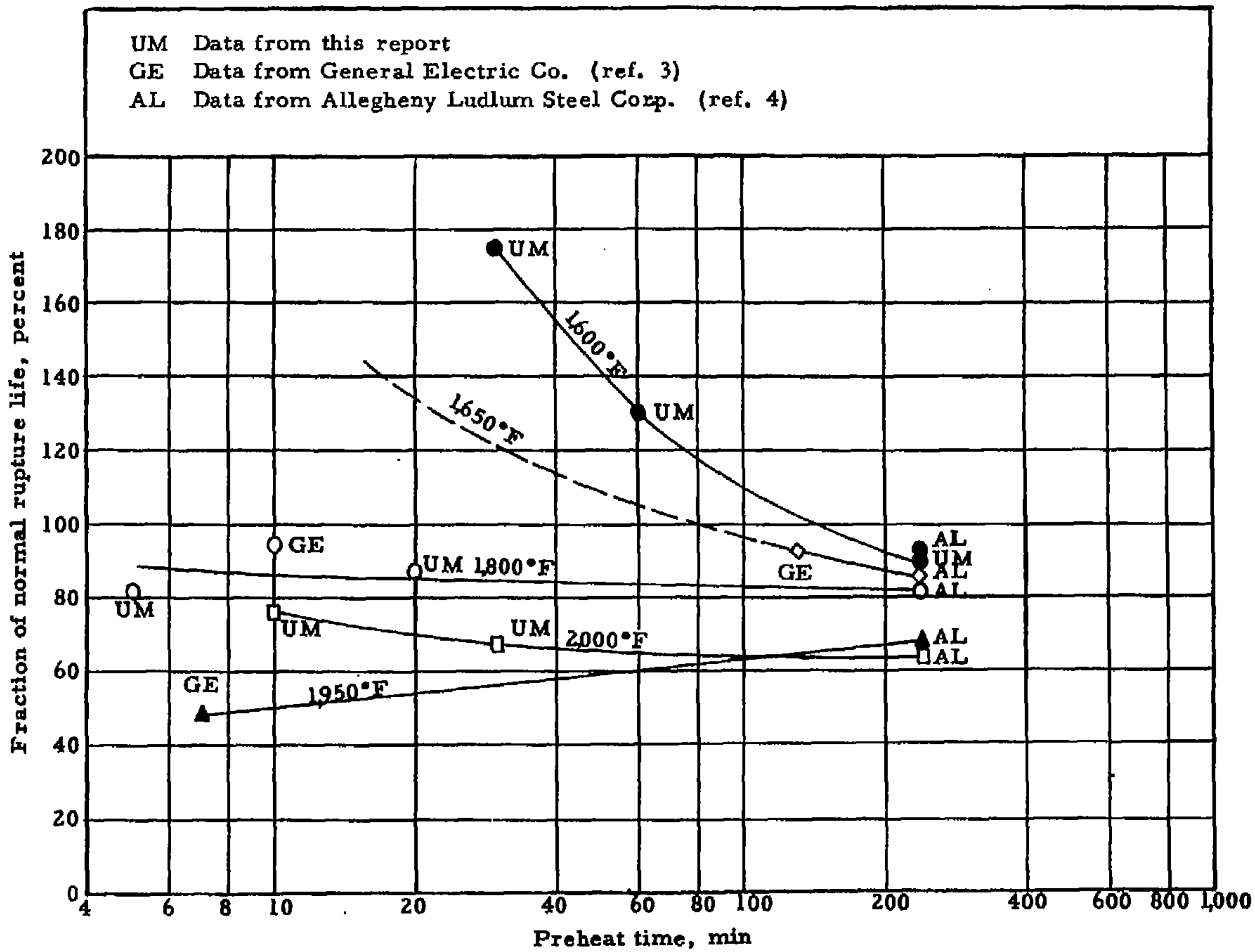


Figure 9.- Change in rupture time due to overheating to 1,600°, 1,650°, 1,800°, 1,950°, and 2,000° F prior to testing.

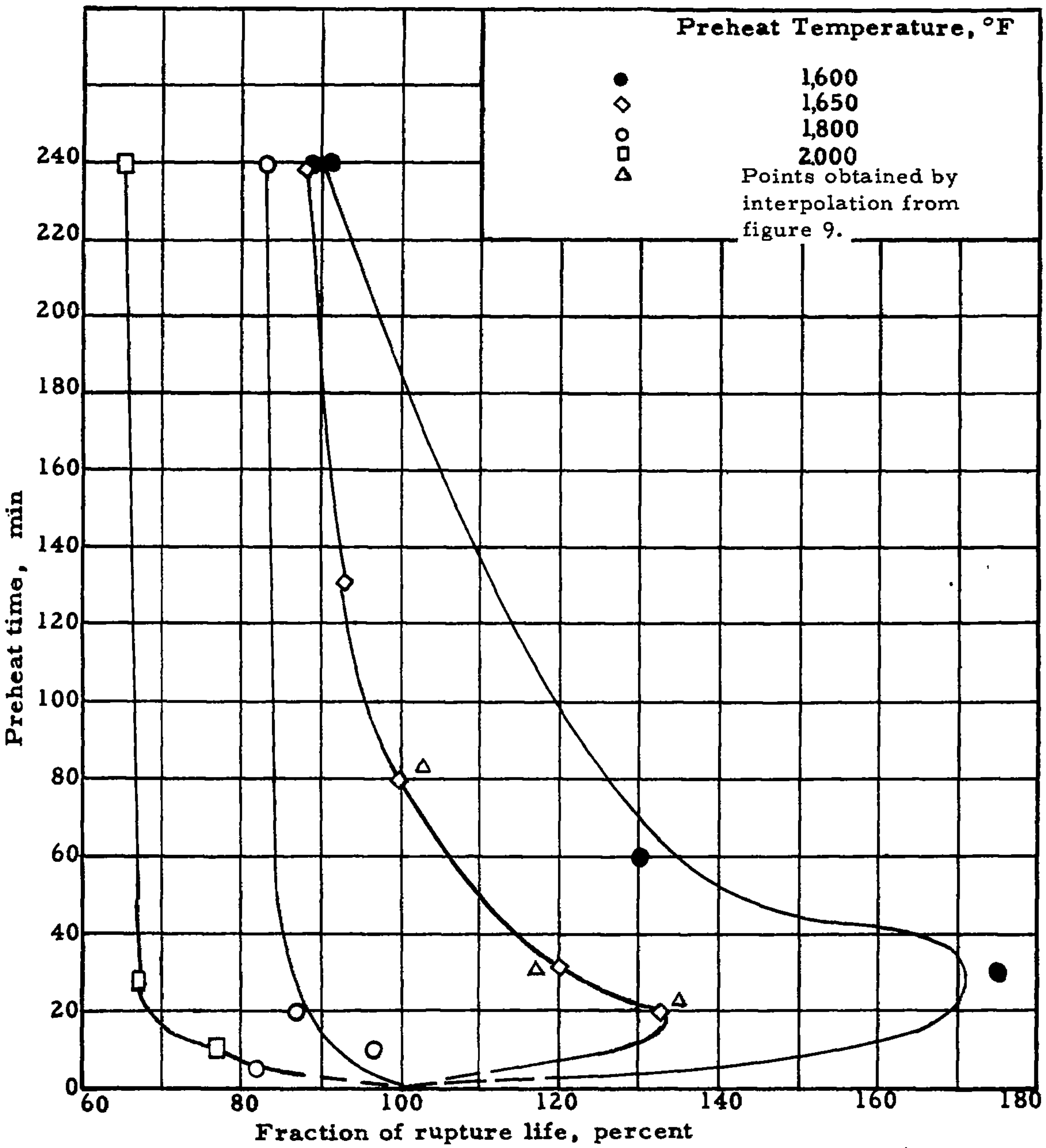
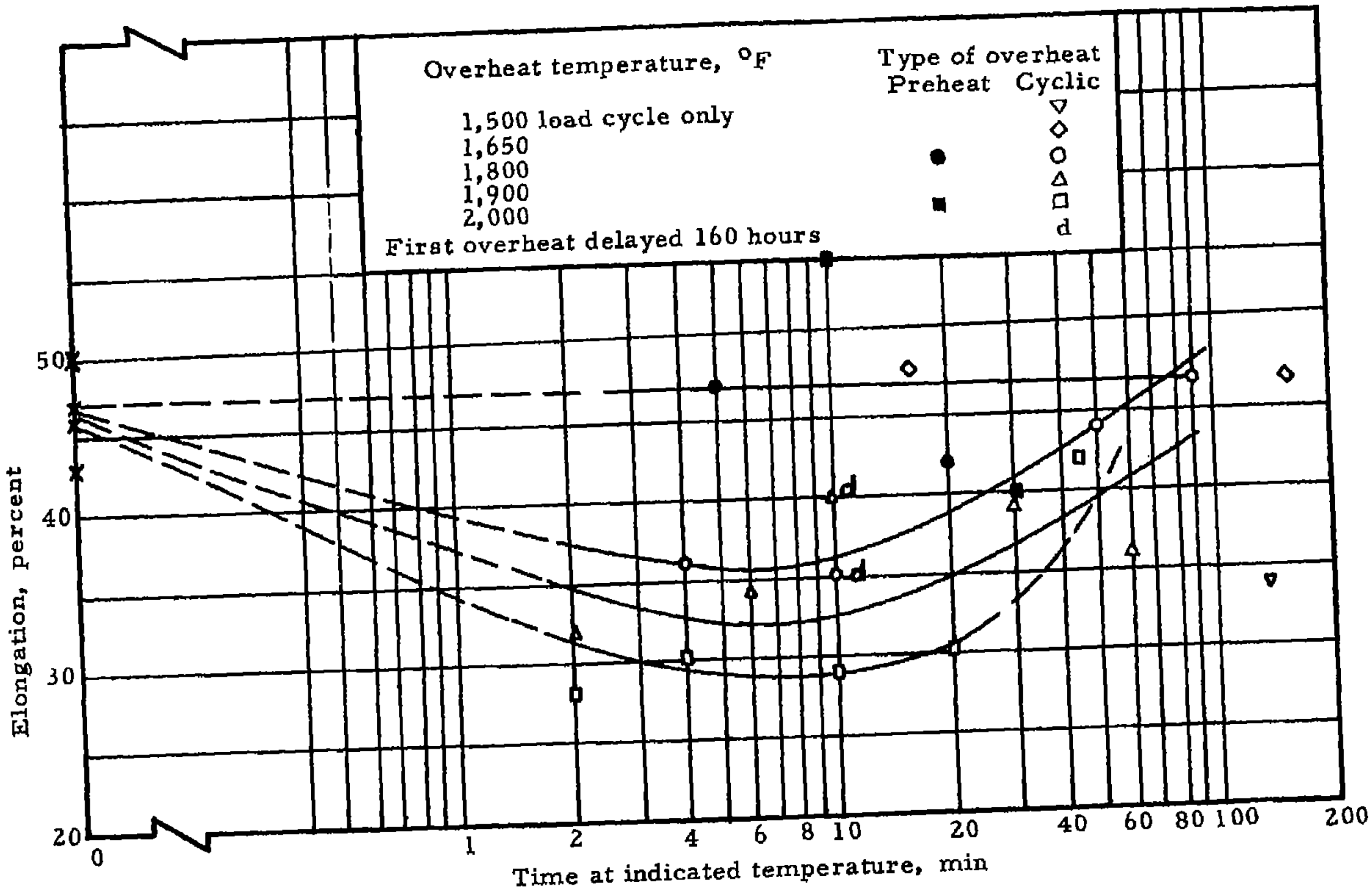
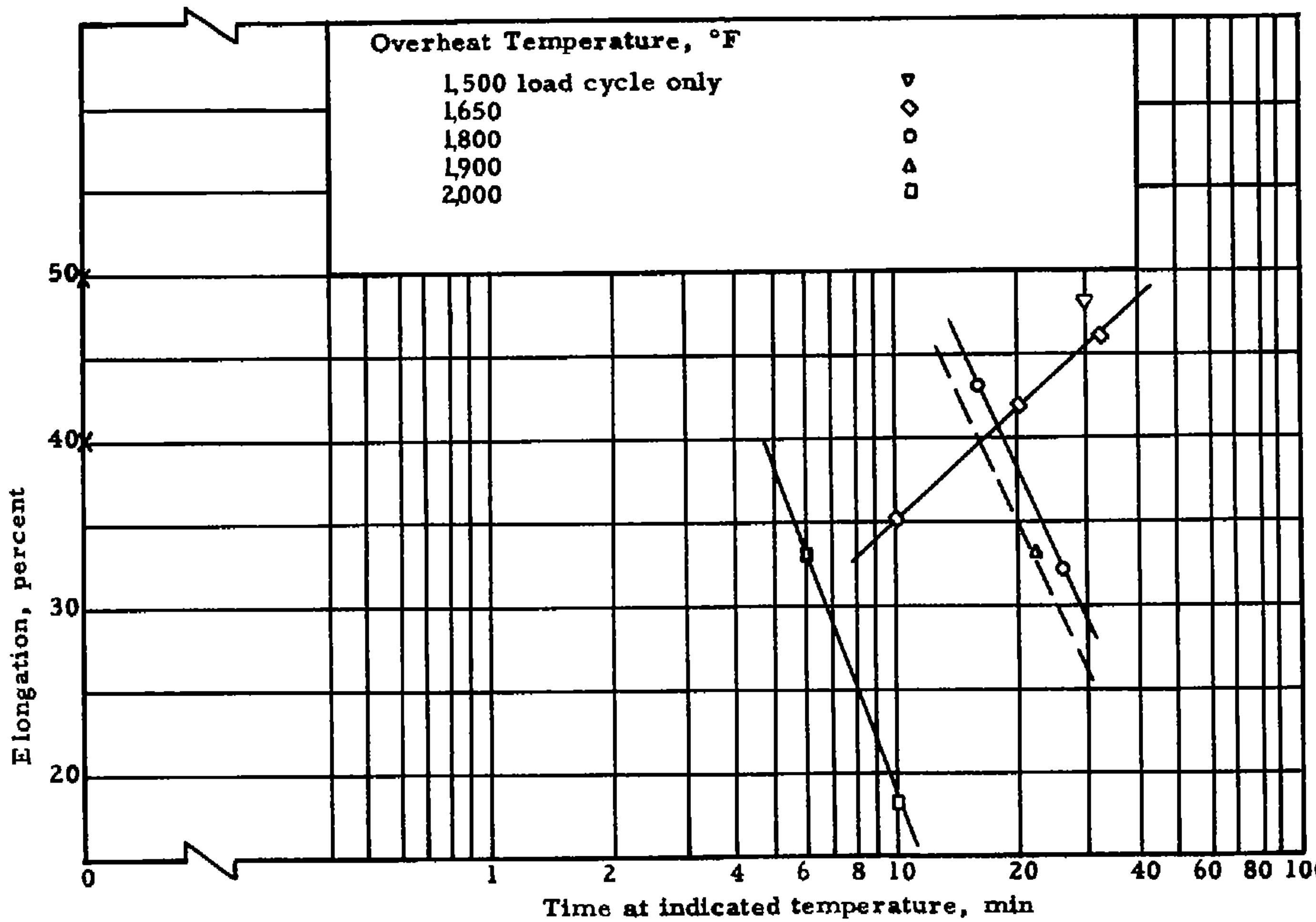


Figure 10.- Percent of rupture life left at 1,500° F after preheating for various times to indicated temperatures.



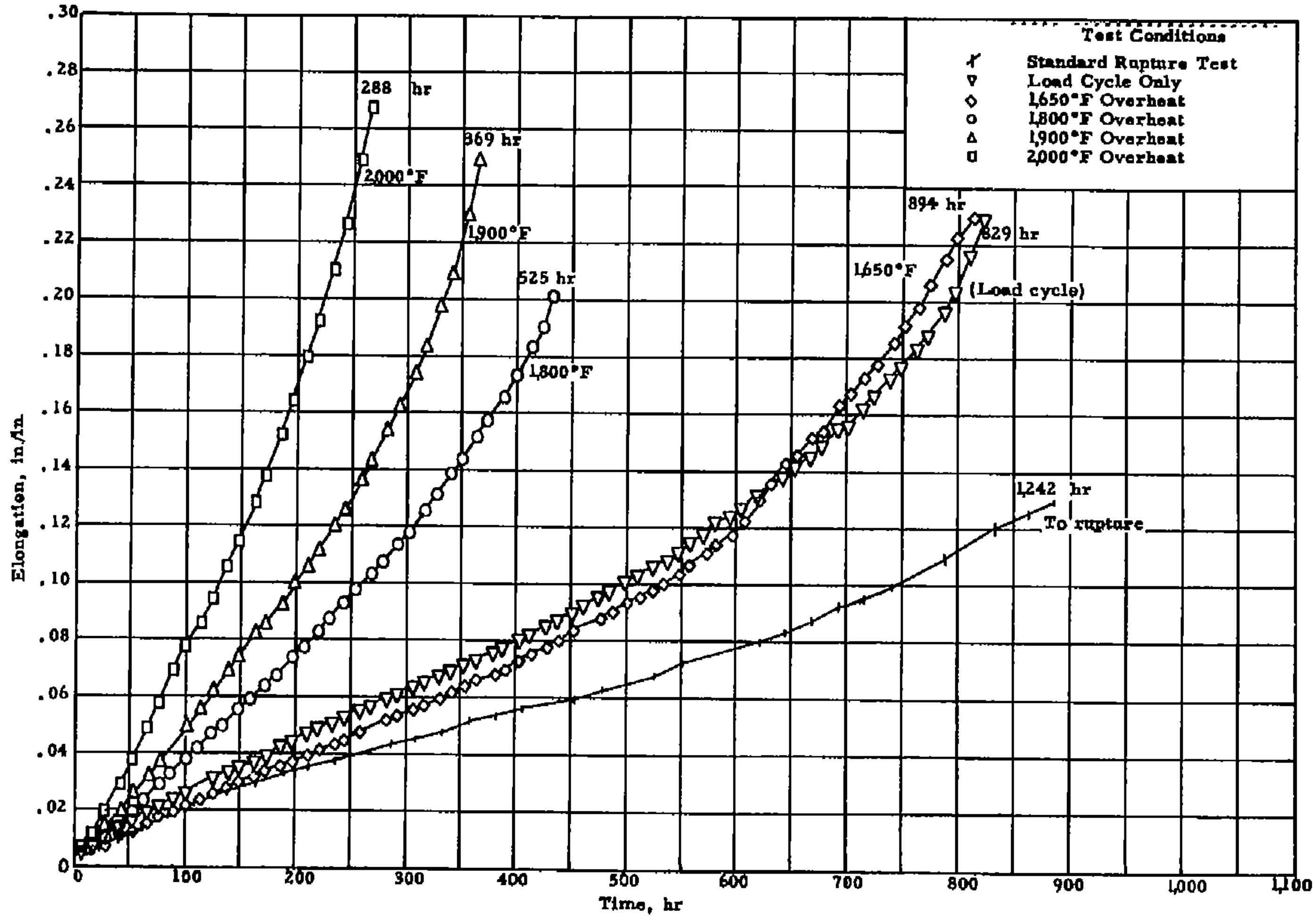
(a) 16,200-psi stress. Cyclic tests received one 2-minute overheat every 12 hours in absence of stress.

Figure 11.- Effect of time at indicated temperature on elongation at rupture under 1,500° F and 16,200 and 22,000 psi. Points on zero axis correspond to data in table I.



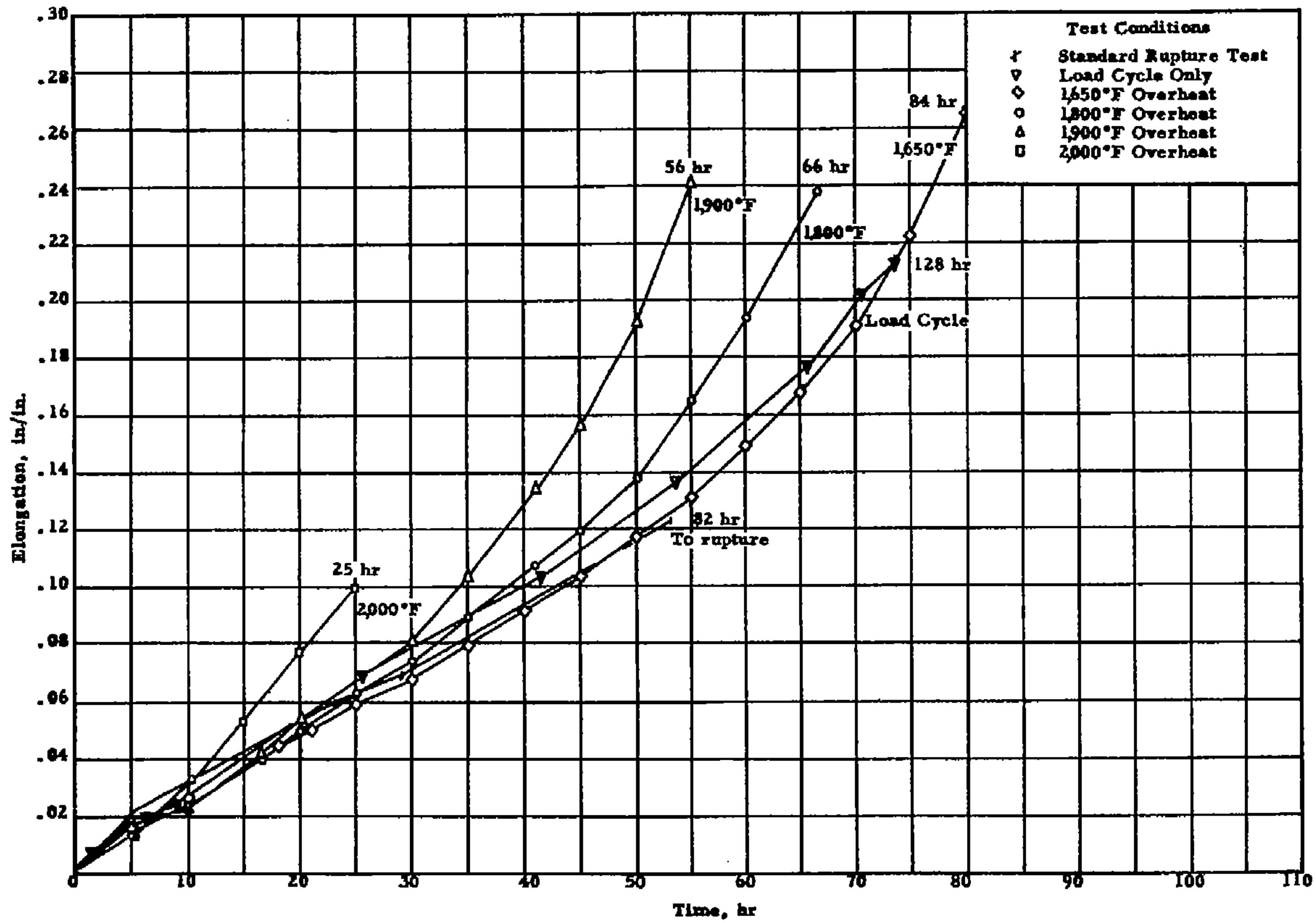
(b) 22,000-psi stress. Tests received one 2-minute overheat every 5 hours in absence of stress.

Figure 11.- Concluded.



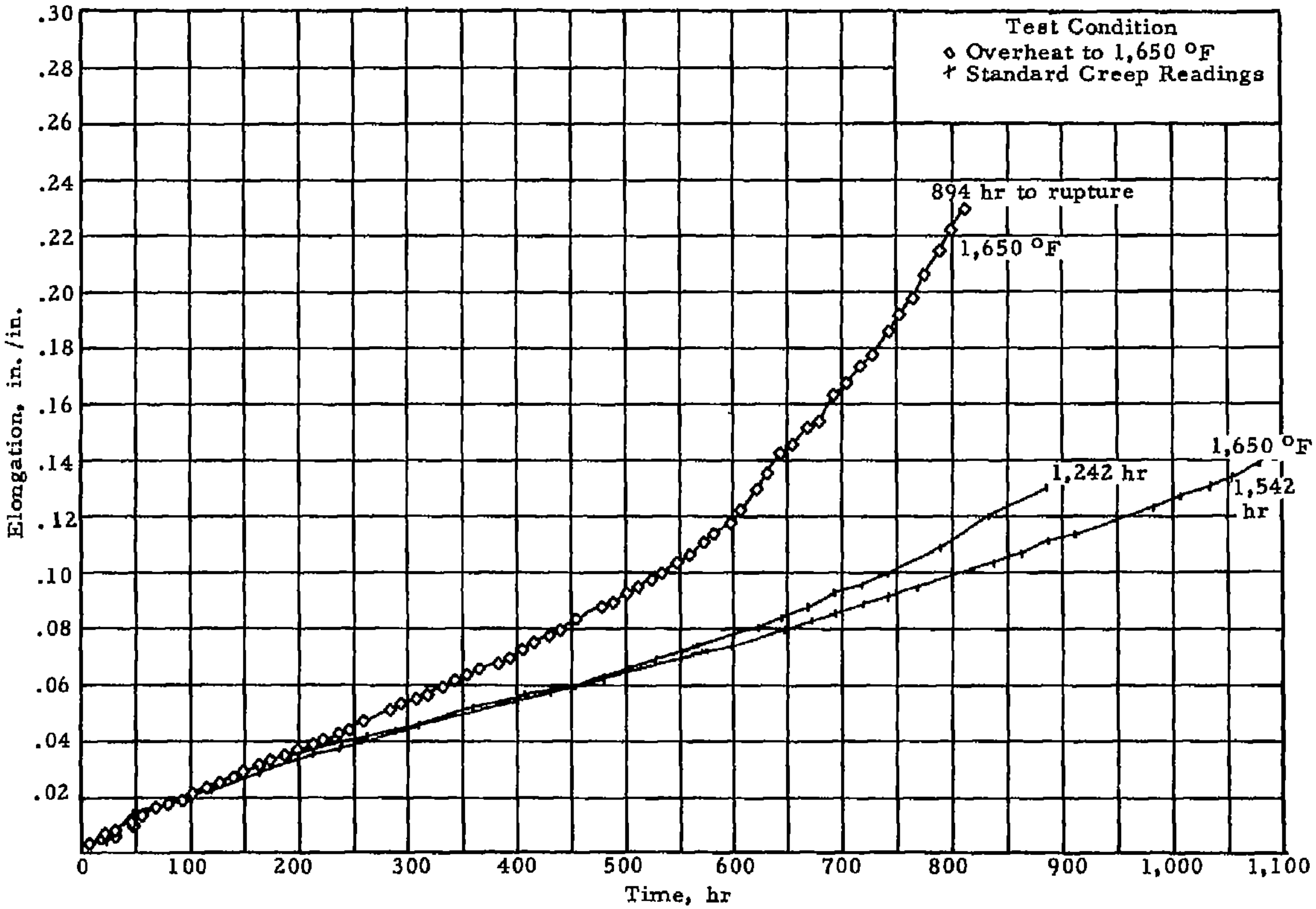
(a) 16,200-psi stress; overheats every 12 hours.

Figure 12.- Comparative creep curves for cyclic overheat tests in absence of stress at 1,500° F and 16,200 and 22,000 psi using 2-minute overheats to indicated temperatures until rupture. Numbers indicate rupture times and overheat temperatures.



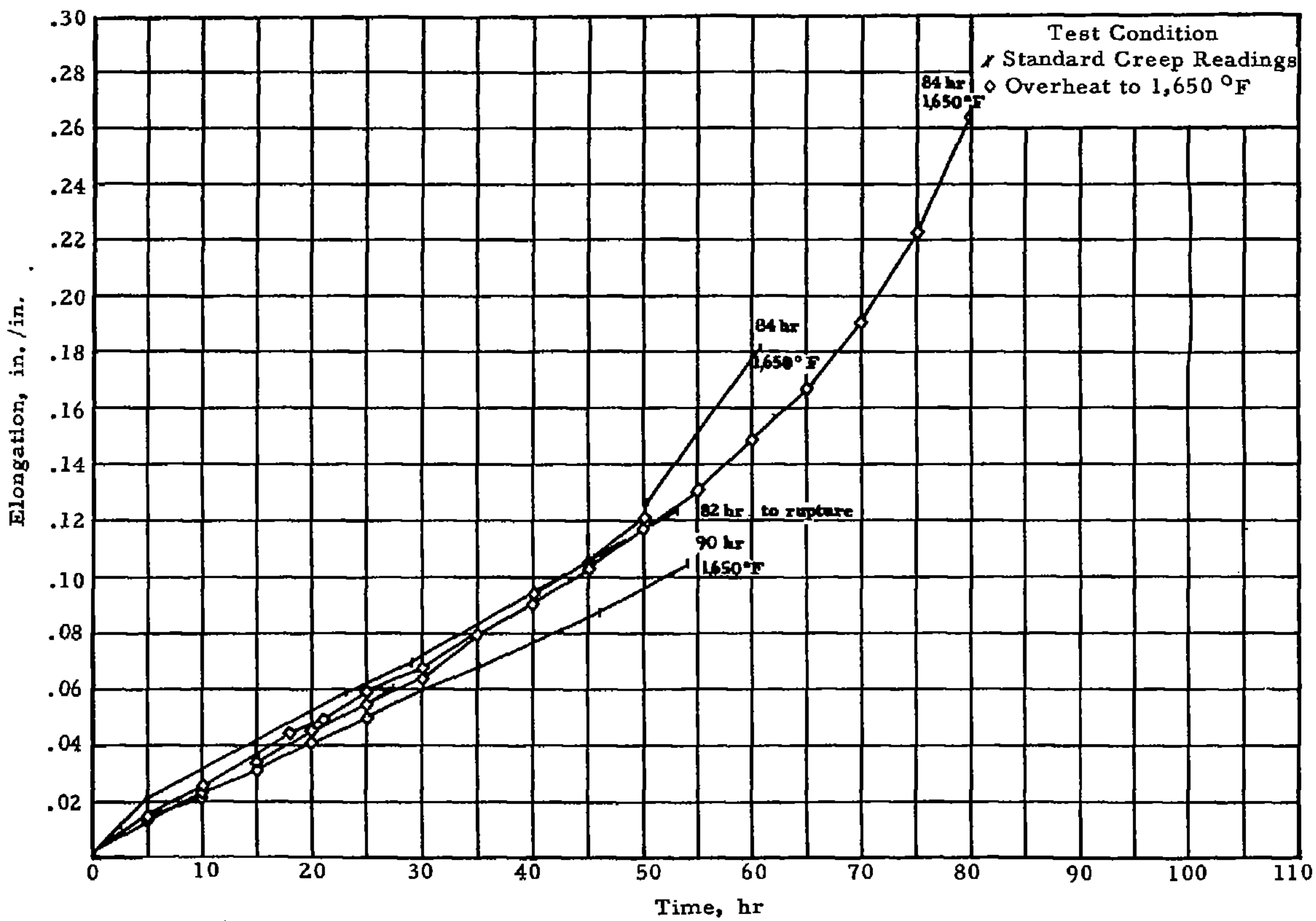
(b) 22,000-psi stress; overheats every 5 hours.

Figure 12.- Concluded.



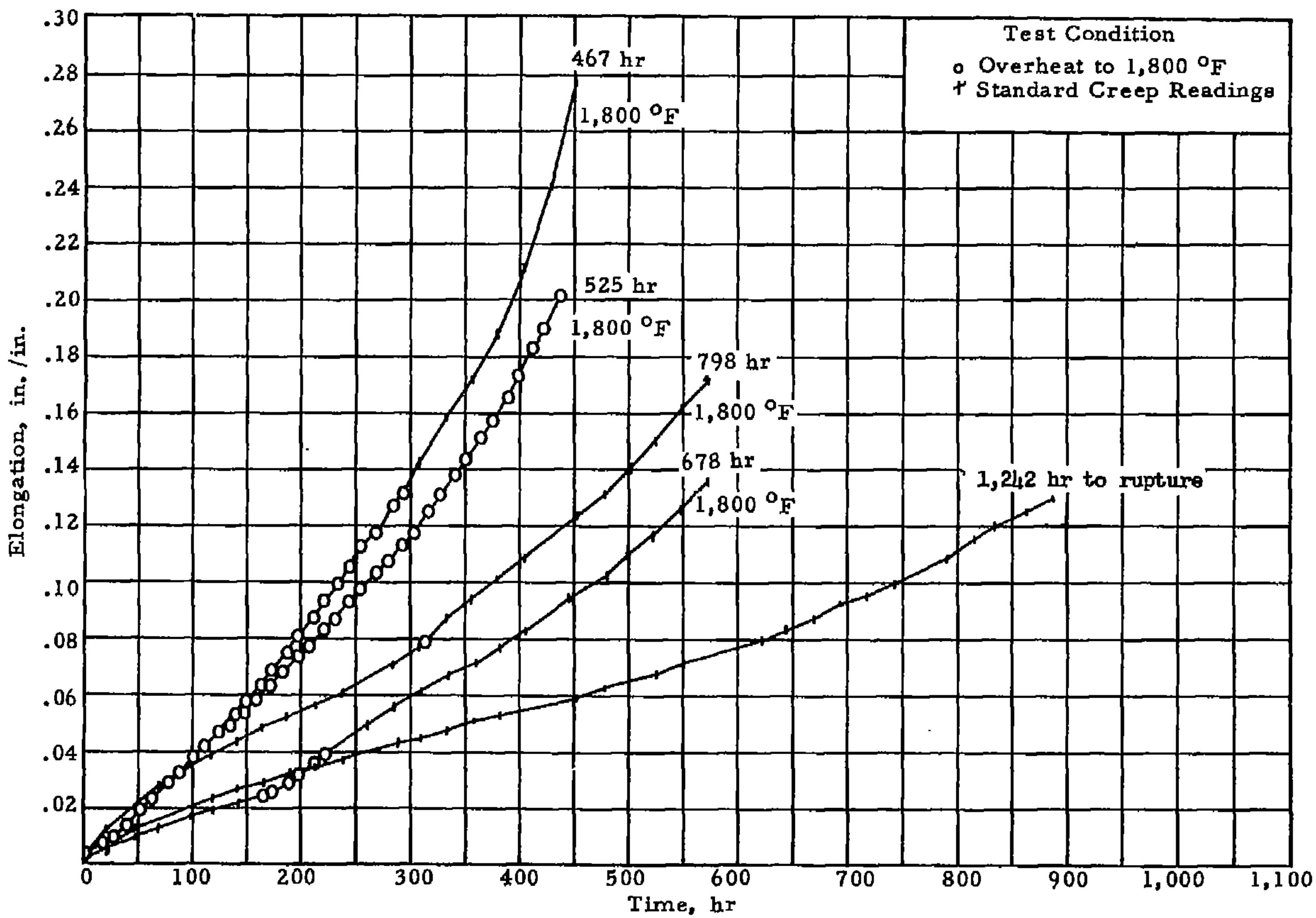
(a) 16,200-psi stress; overheats to 1,650° F.

Figure 13.- Comparative creep curves at 1,500° F and 16,200 and 22,000 psi for tests with limited overheats to various temperatures in absence of stress. Numbers indicate rupture times and overheat temperatures.



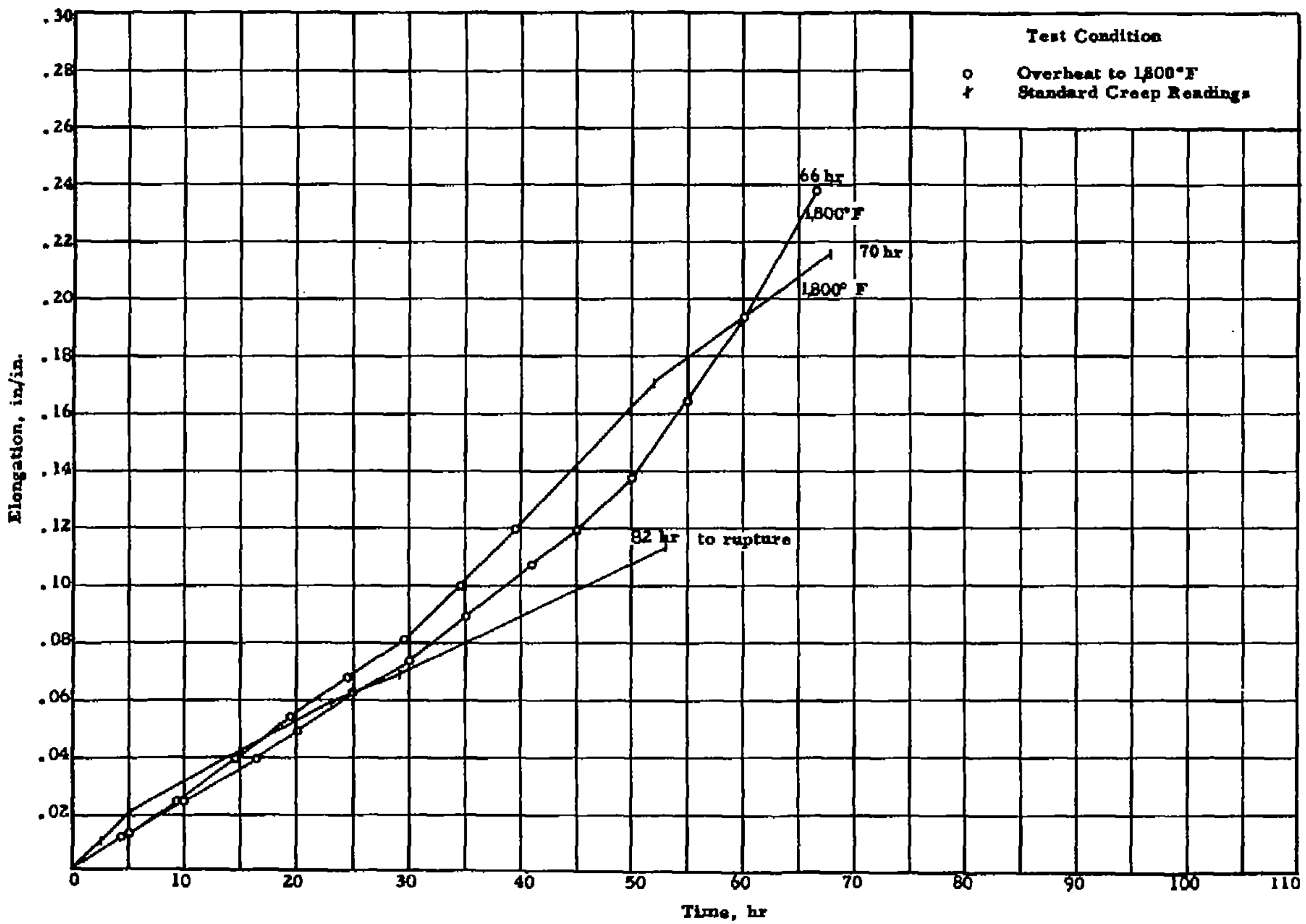
(b) 22,000-psi stress; overheats to 1,650° F.

Figure 13.- Continued.



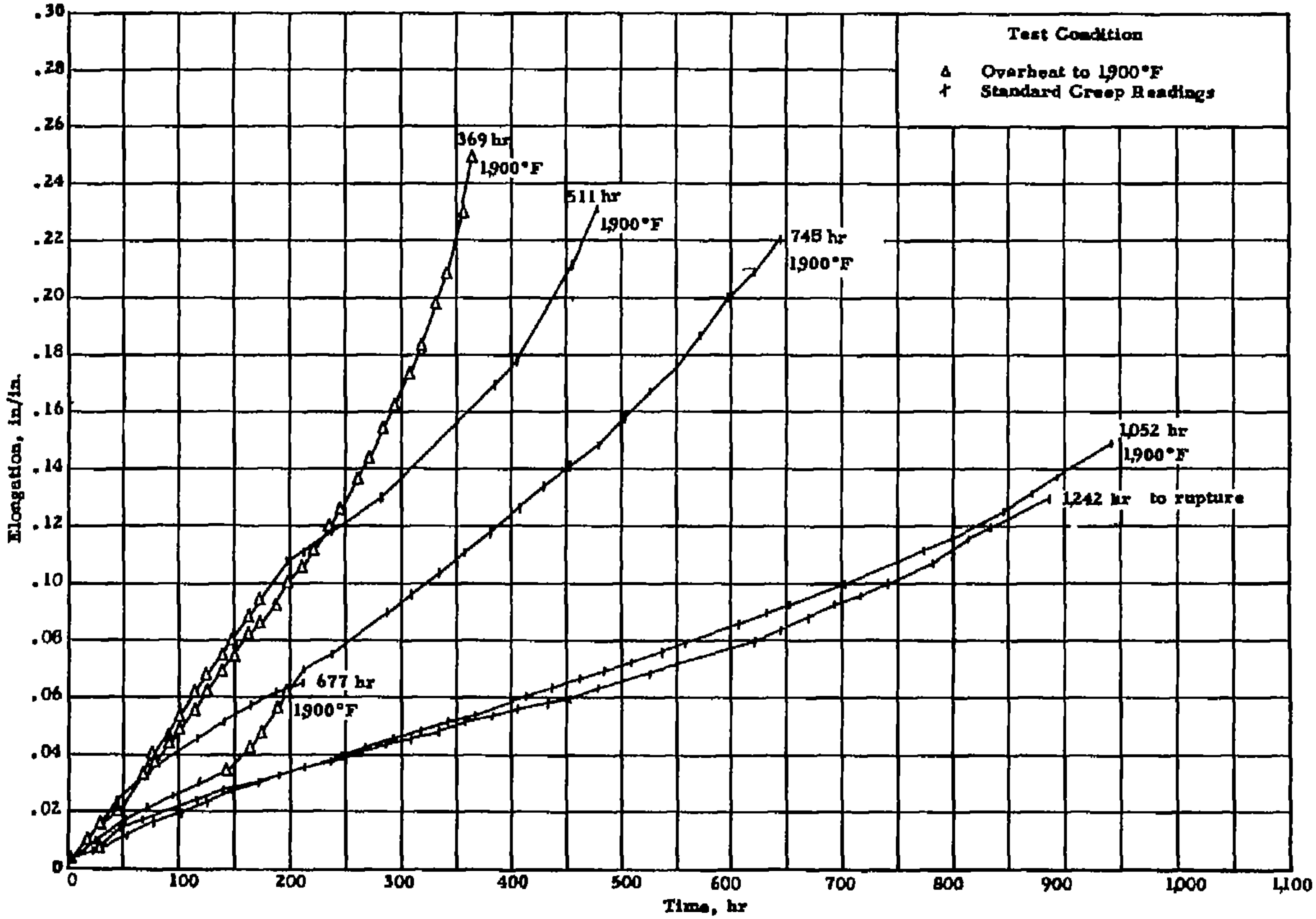
(c) 16,200-psi stress; overheats to 1,800° F.

Figure 13.- Continued.



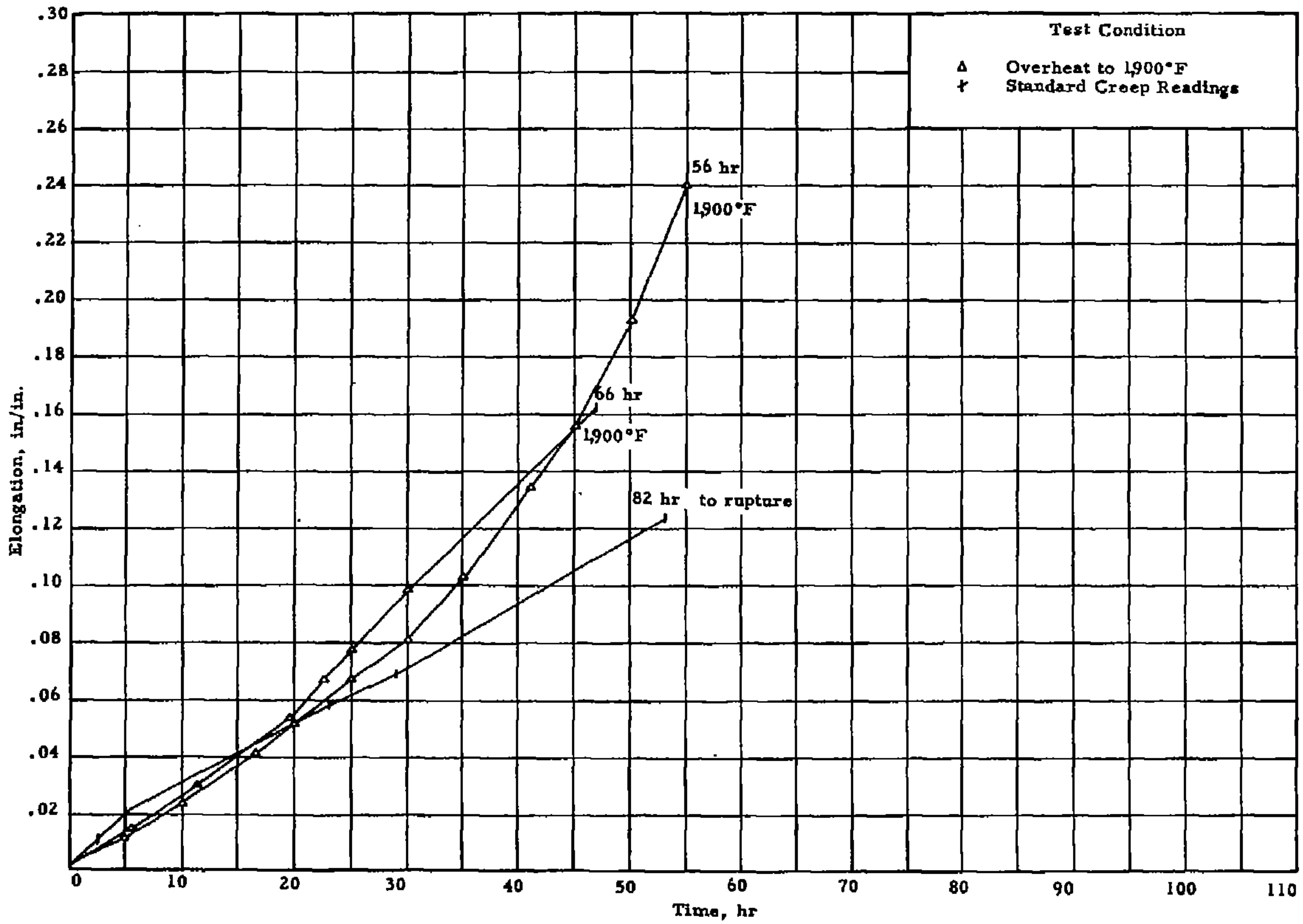
(d) 22,000-psi stress; overheats to 1,800° F.

Figure 13.- Continued.



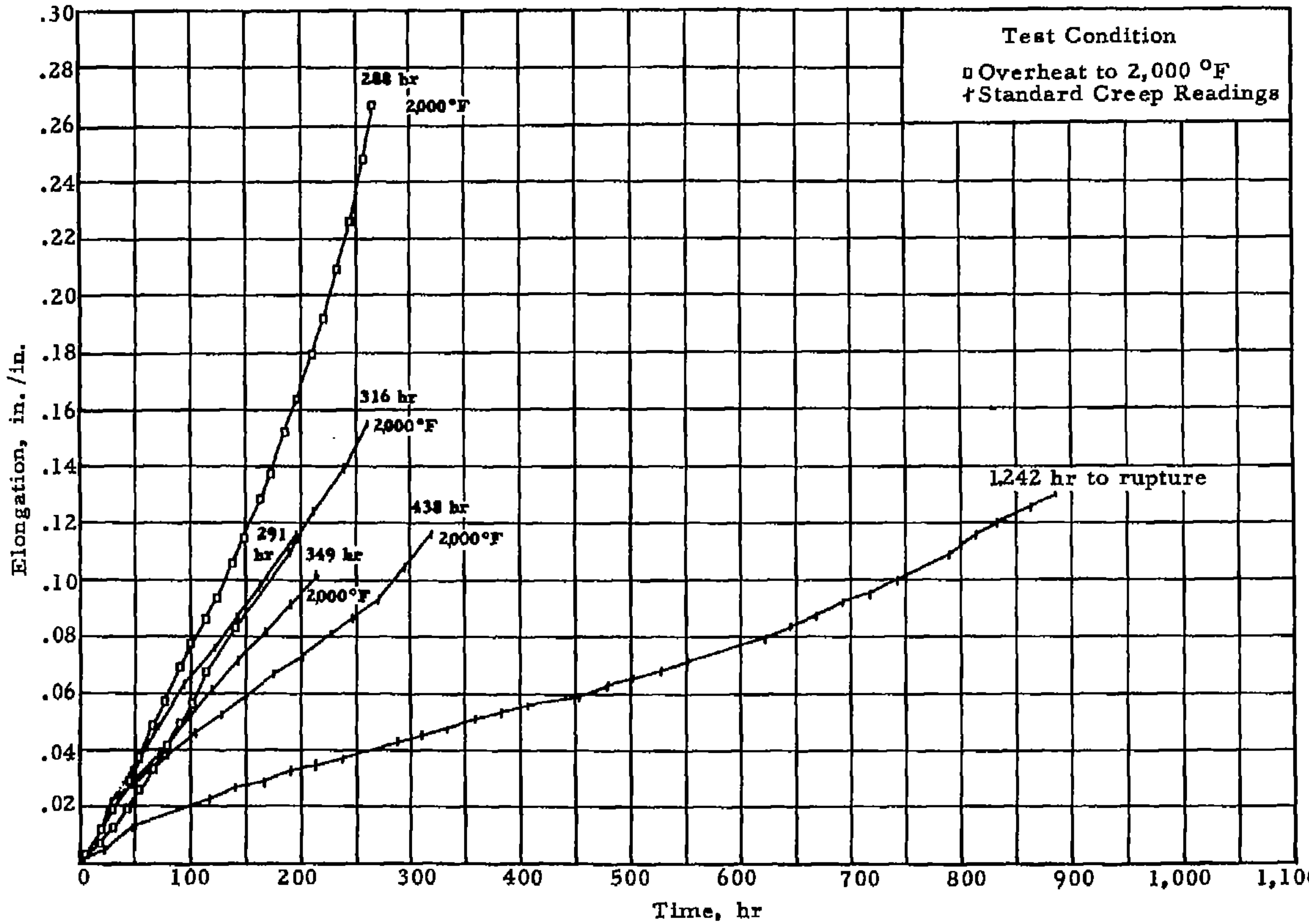
(e) 16,200-psi stress; overheats to 1,900° F.

Figure 13.- Continued.



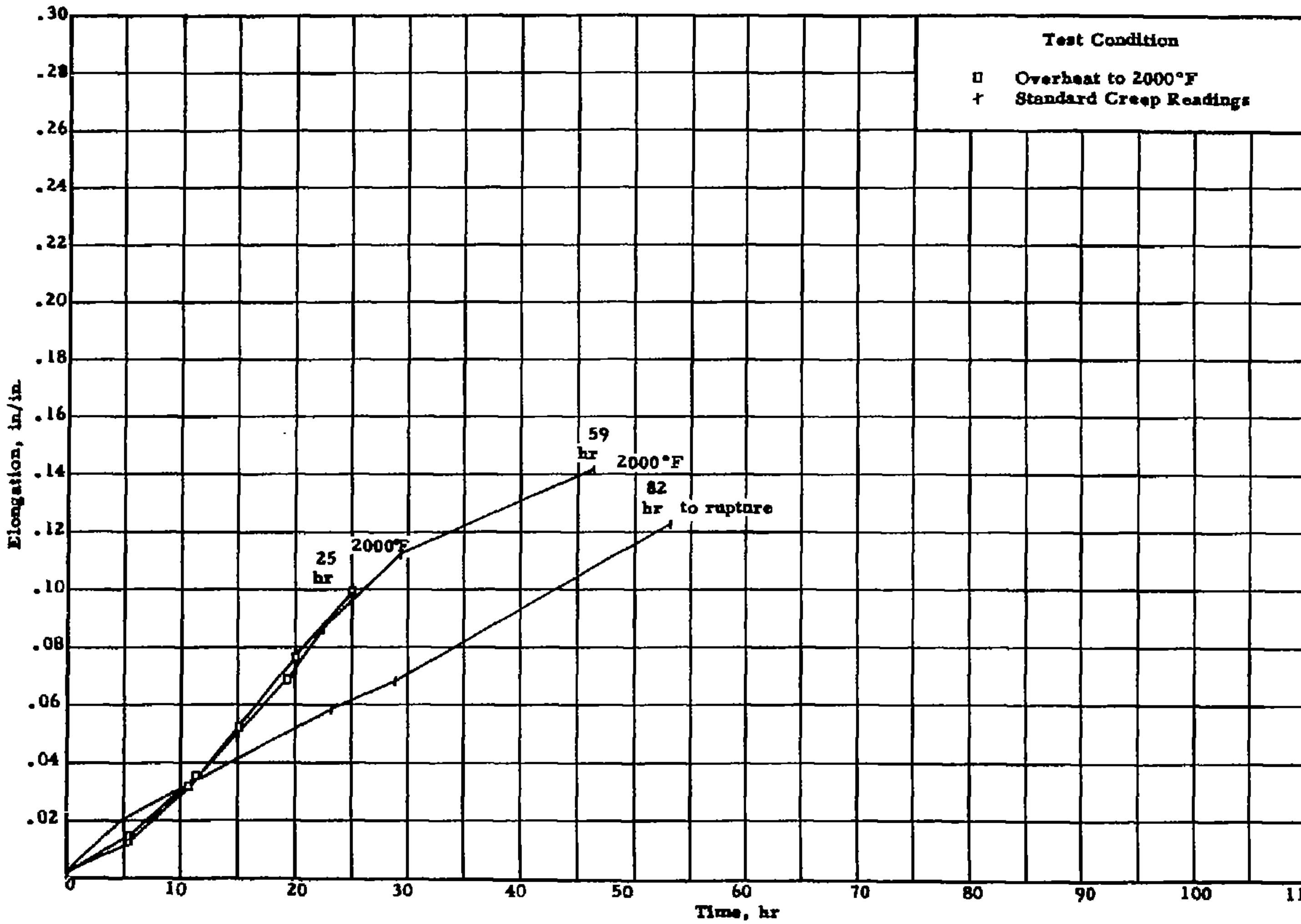
(f) 22,000-psi stress; overheats to 1,900° F.

Figure 13.- Continued.



(g) 16,200-psi stress; overheats to 2,000° F.

Figure 13.- Continued.



(h) 22,000-psi stress; overheats to 2,000° F.

Figure 13.- Concluded.

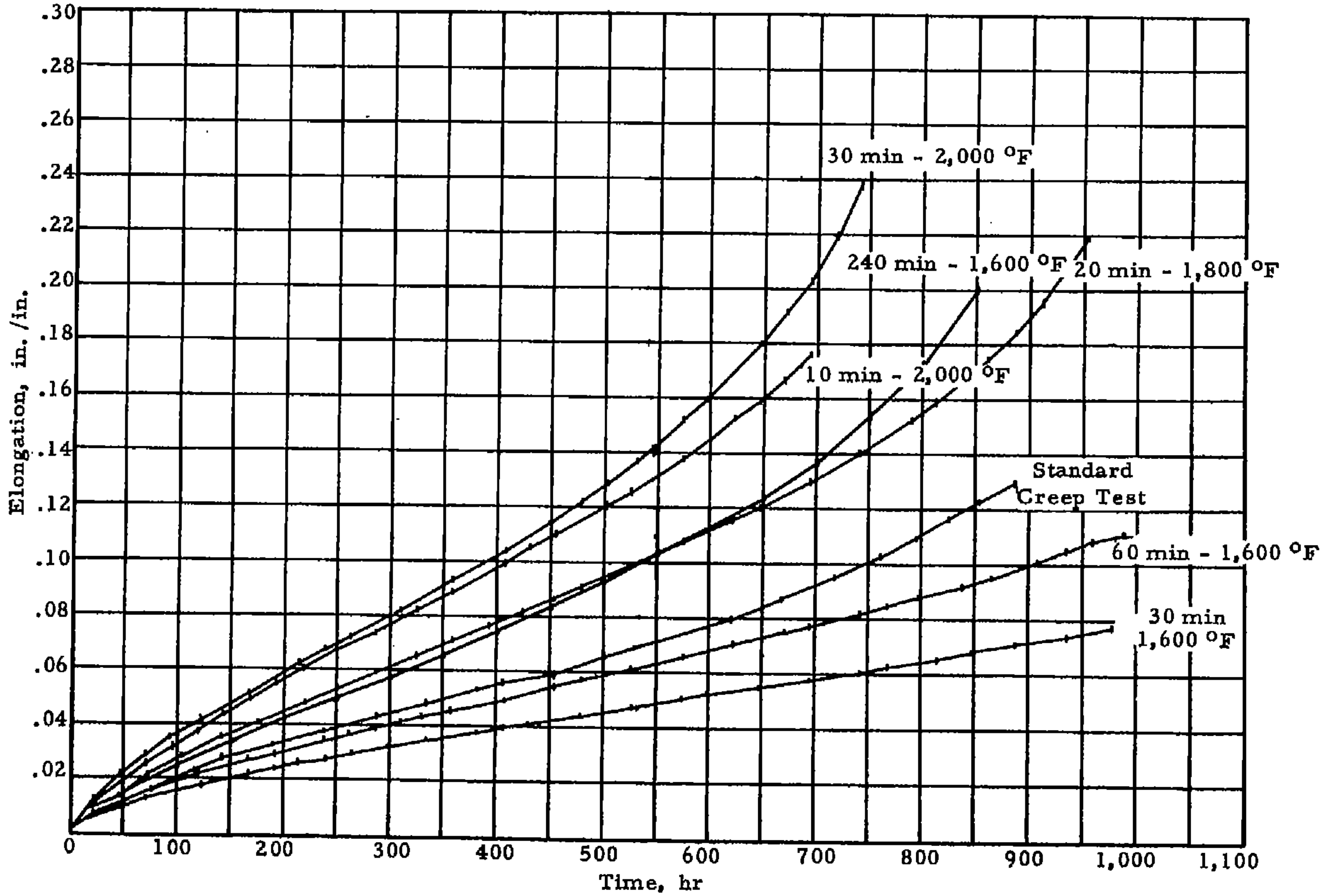


Figure 14.- Comparative creep curves at 1,500° F and 16,200 psi for tests preheated to various temperatures for indicated times.

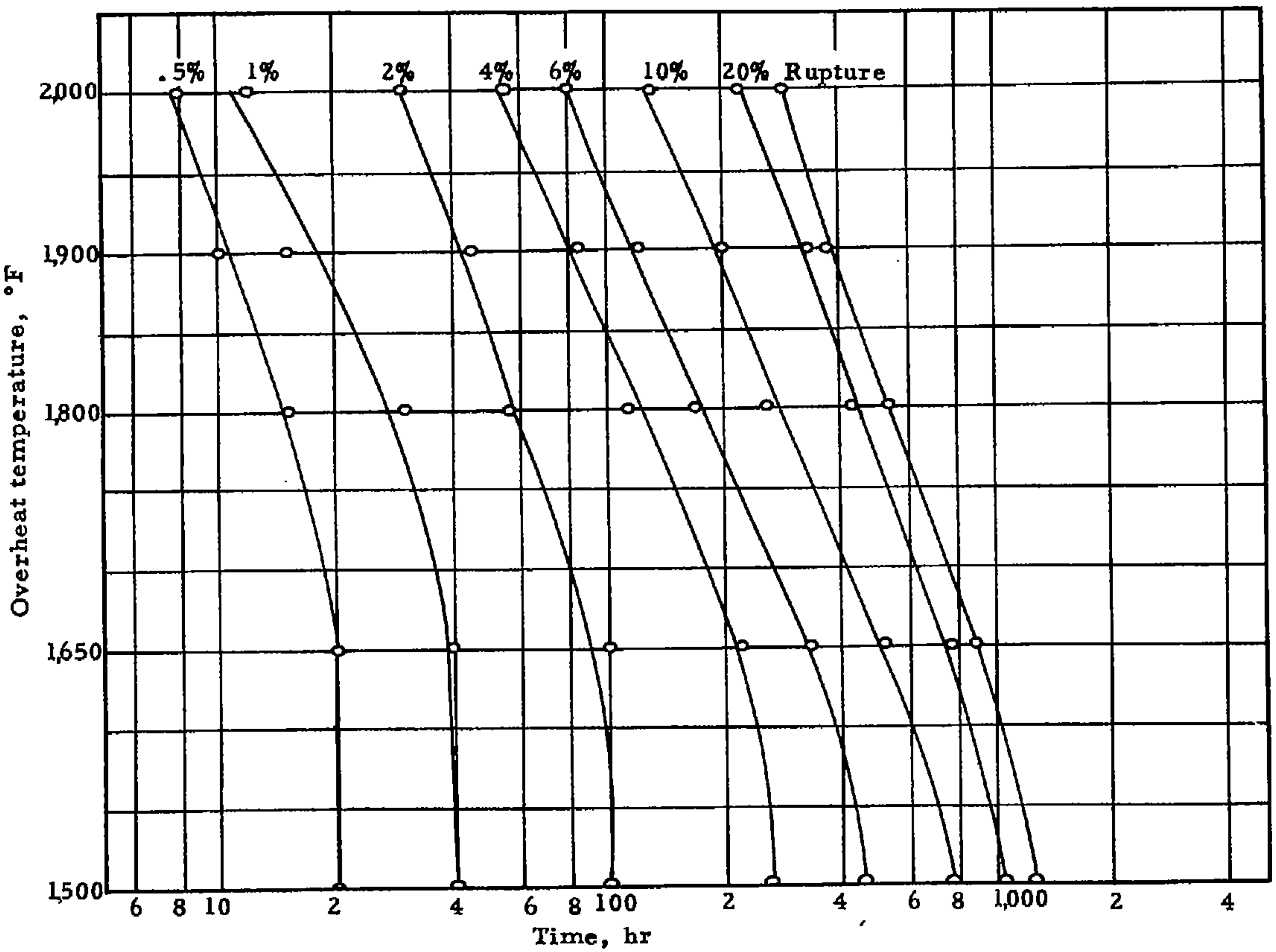
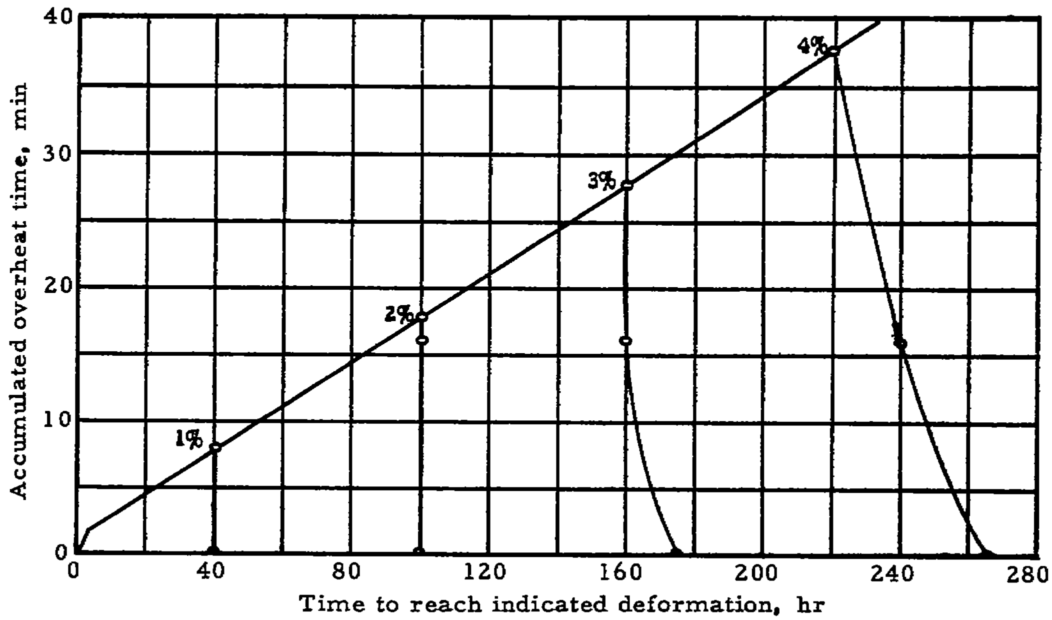
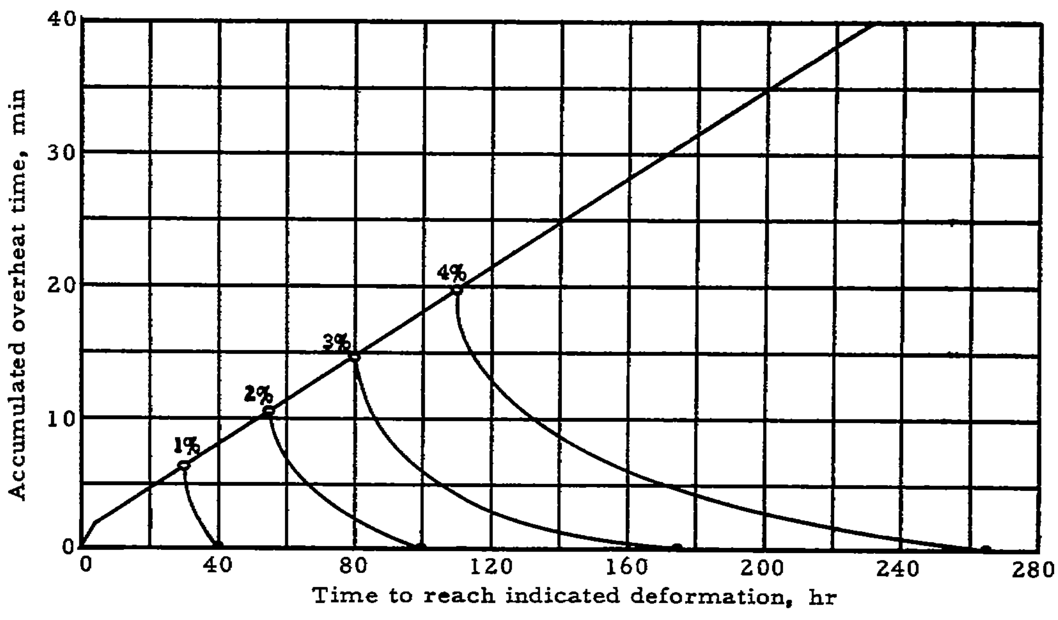


Figure 15.- Influence of temperature of continued cyclic overheating in absence of stress on time to reach indicated total deformation and time for rupture for tests at 1,500° F and 16,200 psi.

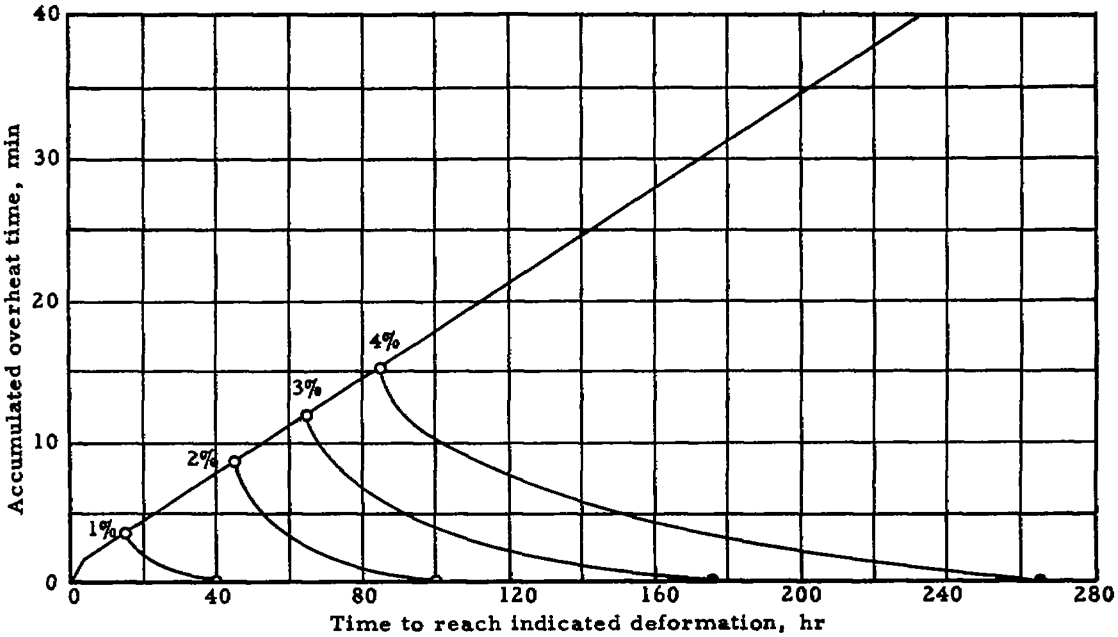


(a) Overheats to 1,650° F.

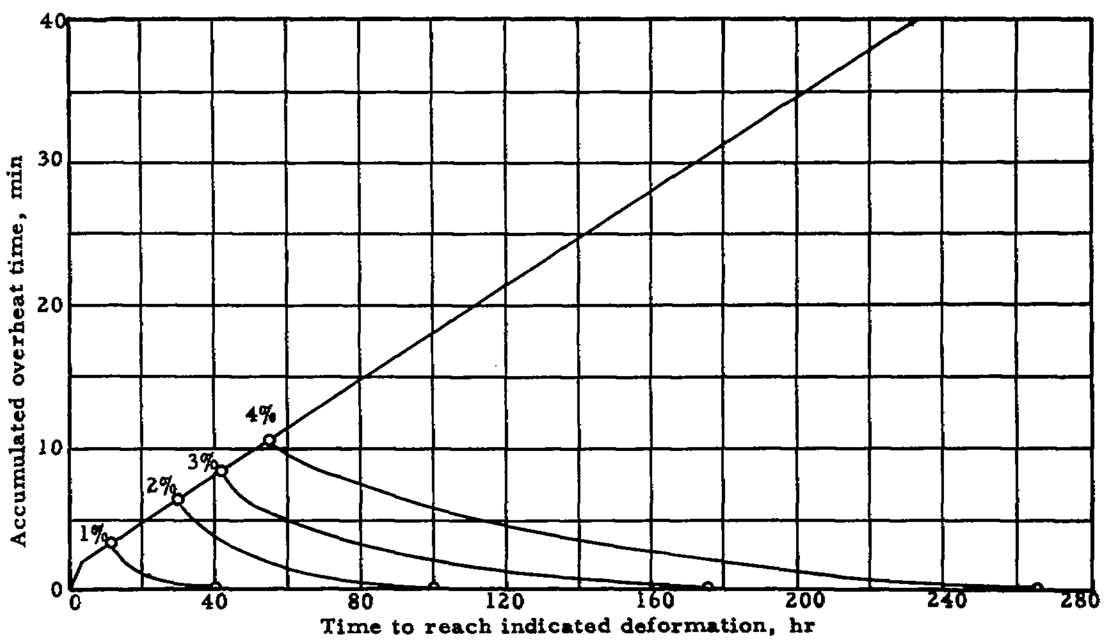


(b) Overheats to 1,800° F.

Figure 16.- Effect of limited overheating on time required to reach indicated total deformation at 1,500° F and 16,200 psi for overheats to various temperatures in absence of stress.



(c) Overheats to 1,900° F.



(d) Overheats to 2,000° F.

Figure 16.- Concluded.

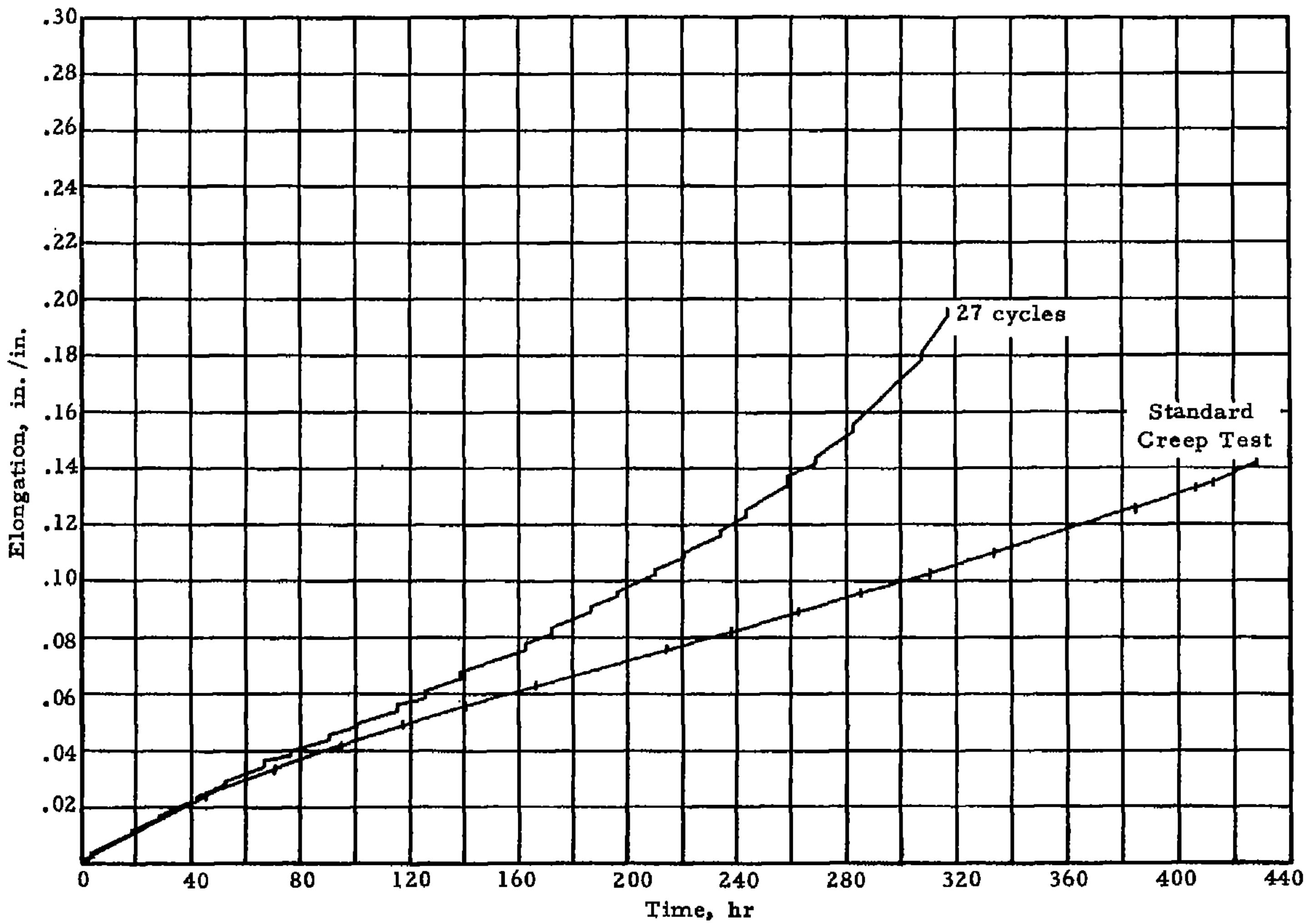


Figure 17.- Creep curve at 1,500° F and 18,000 psi for cyclic overheat test under 18,000 psi to 1,650° F until rupture compared with standard creep curve at these conditions. Overheats of 2 minutes applied every 12 hours.

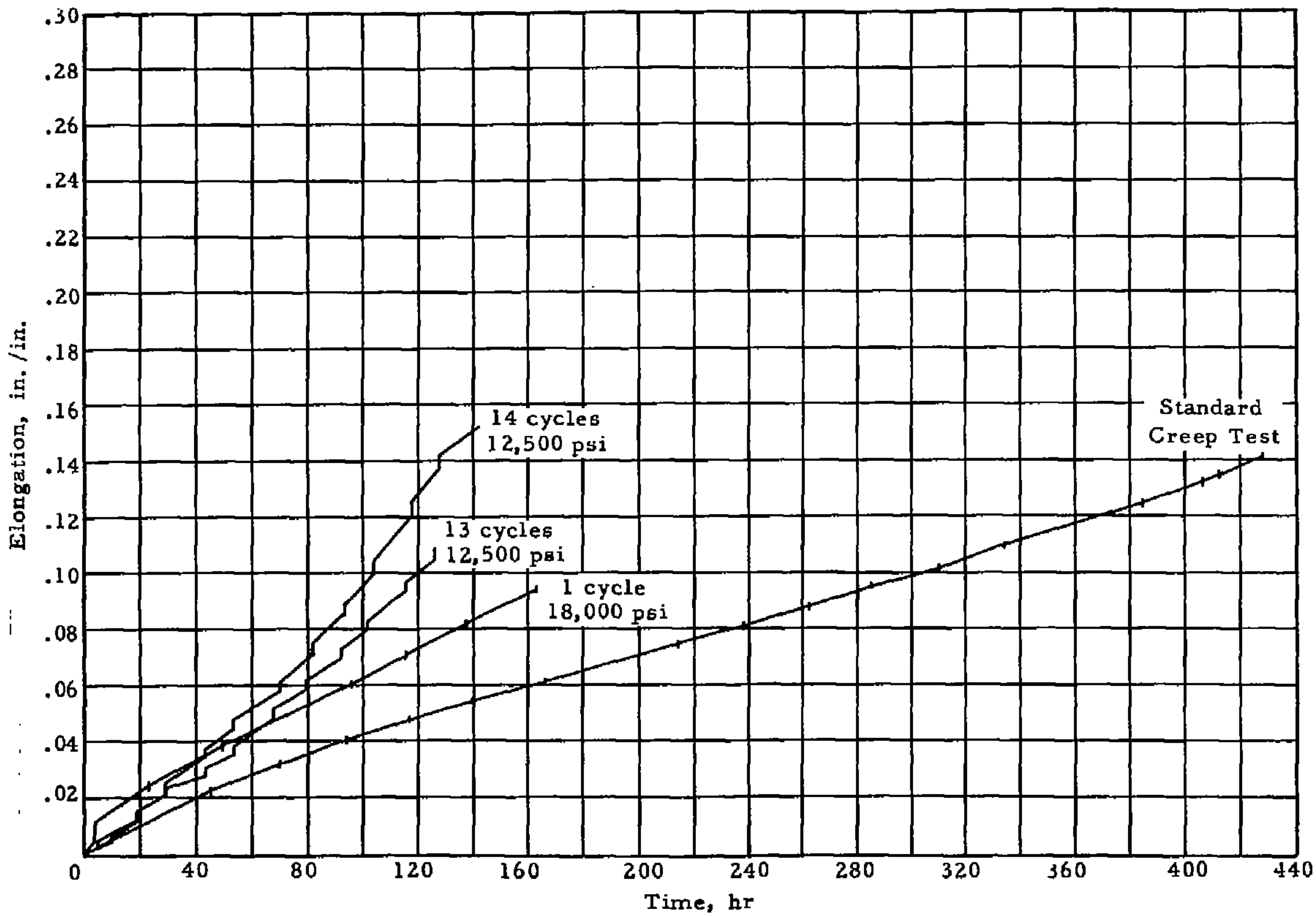
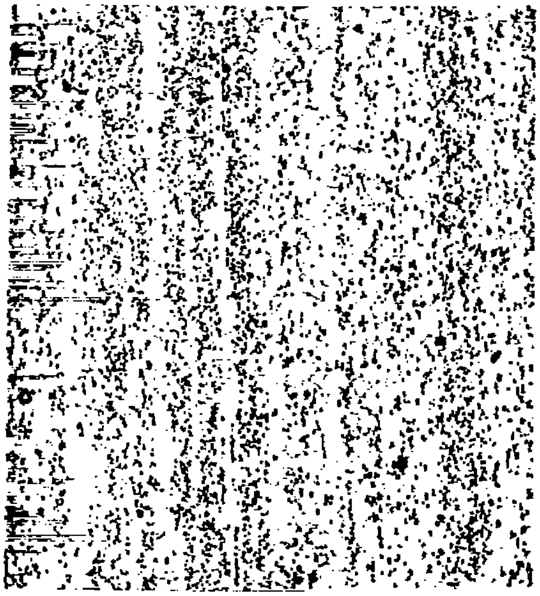
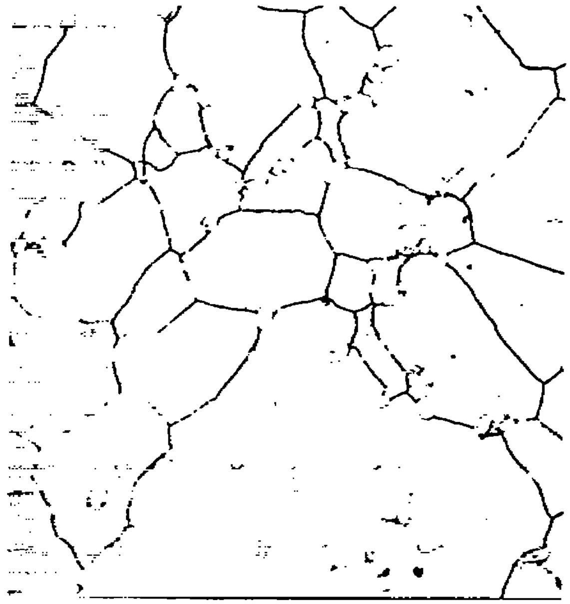


Figure 18.- Comparative creep curves at 1,500° F and 18,000 psi for cyclic overheat tests to 1,800° F under indicated stress for number of cycles shown. Overheats of 2 minutes applied every 12 hours.

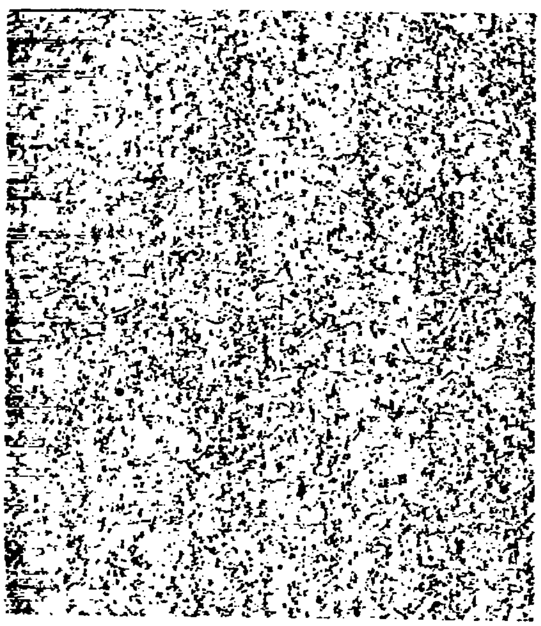


X100



X1000

(a) Rerolled stock after heat treatment.

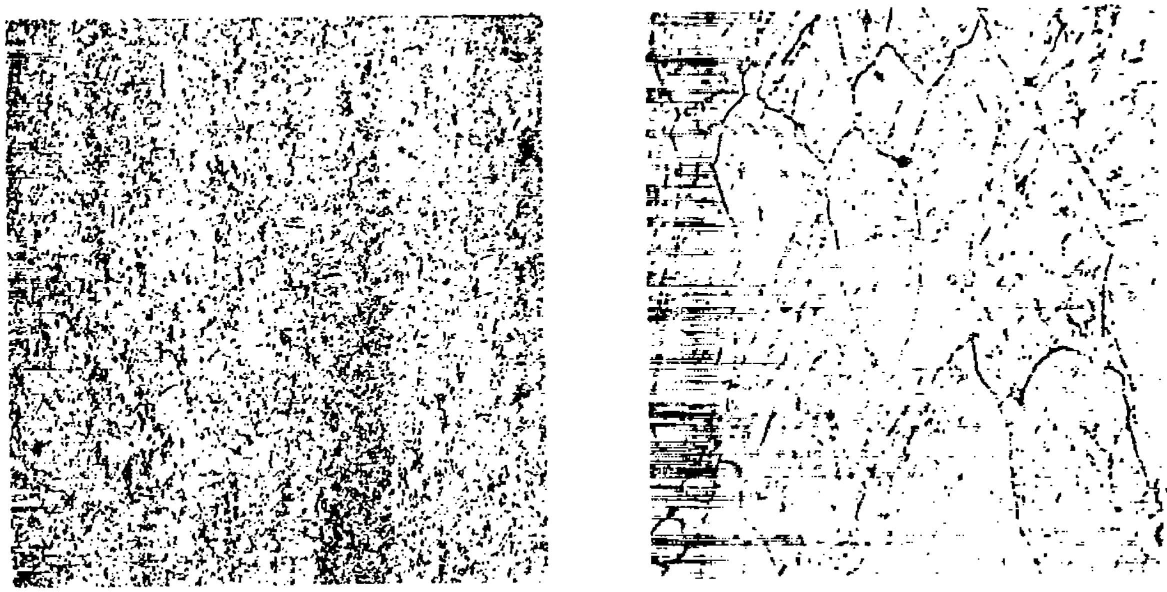


X100

X1000

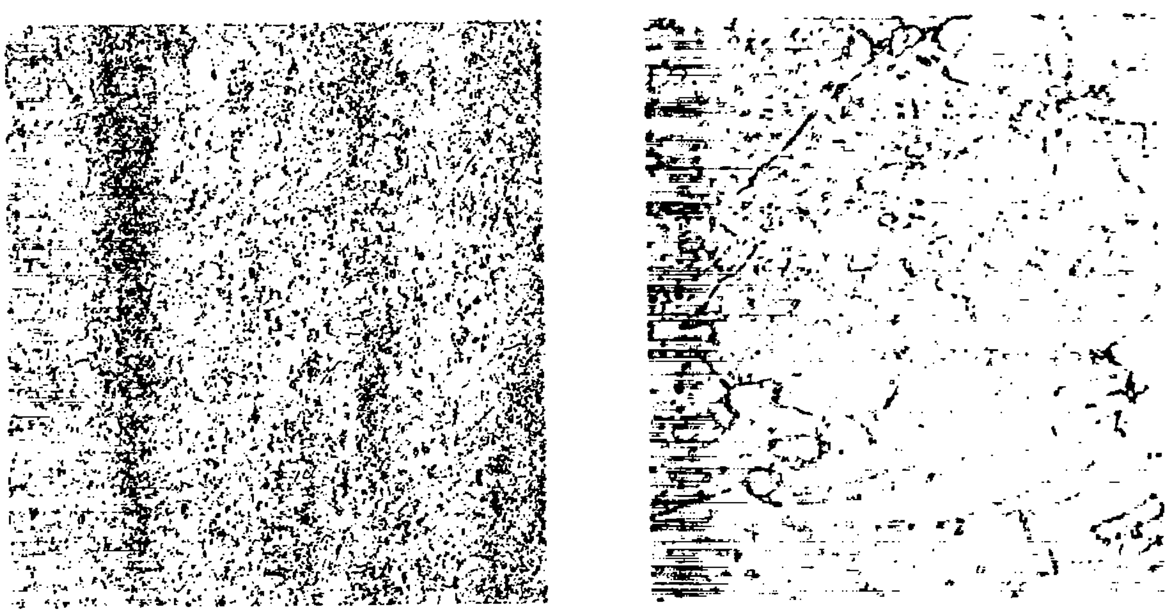
(b) Rerolled stock after rupture-testing at 1,500° F and 16,200 psi (1,242 hours).

Figure 19.- Microstructure of rerolled material after heat treatment and after rupture-testing.



X100 X1000

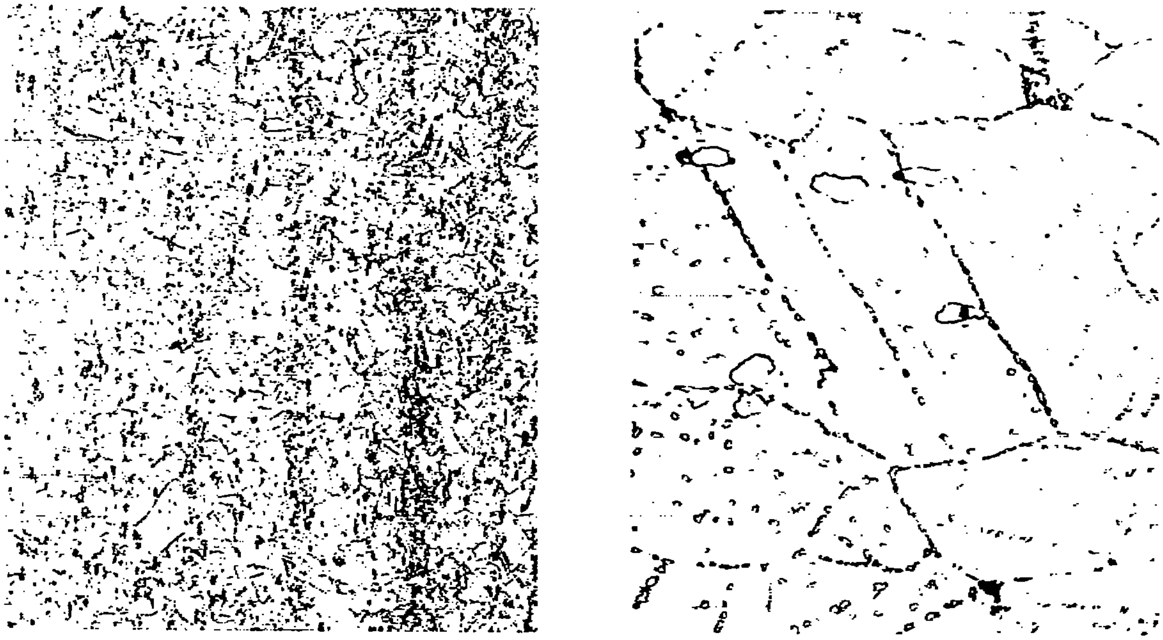
(a) 76 overheats to 1,650° F. Rupture time, 894.2 hours.



X100 X1000

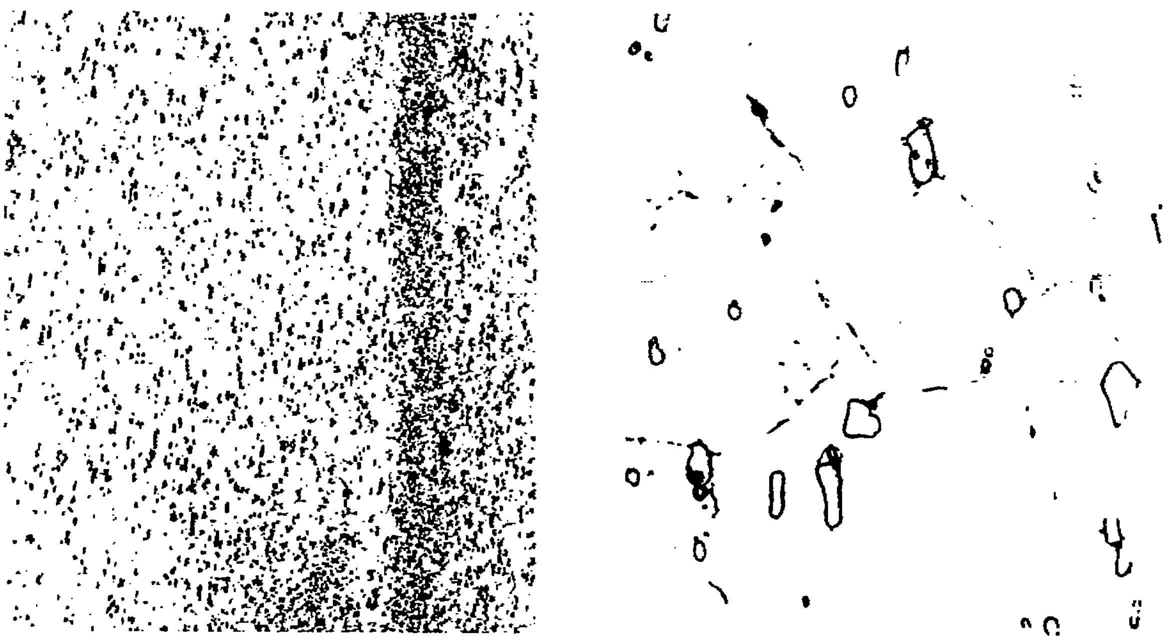
(b) 44 overheats to 1,800° F. Rupture time, 524.5 hours.

Figure 20.- Effect of cyclic overheating until rupture on microstructure of specimens tested at 1,500° F and 16,200 psi. Stress removed during 2-minute overheat cycles every 12 hours.



X100 X1000

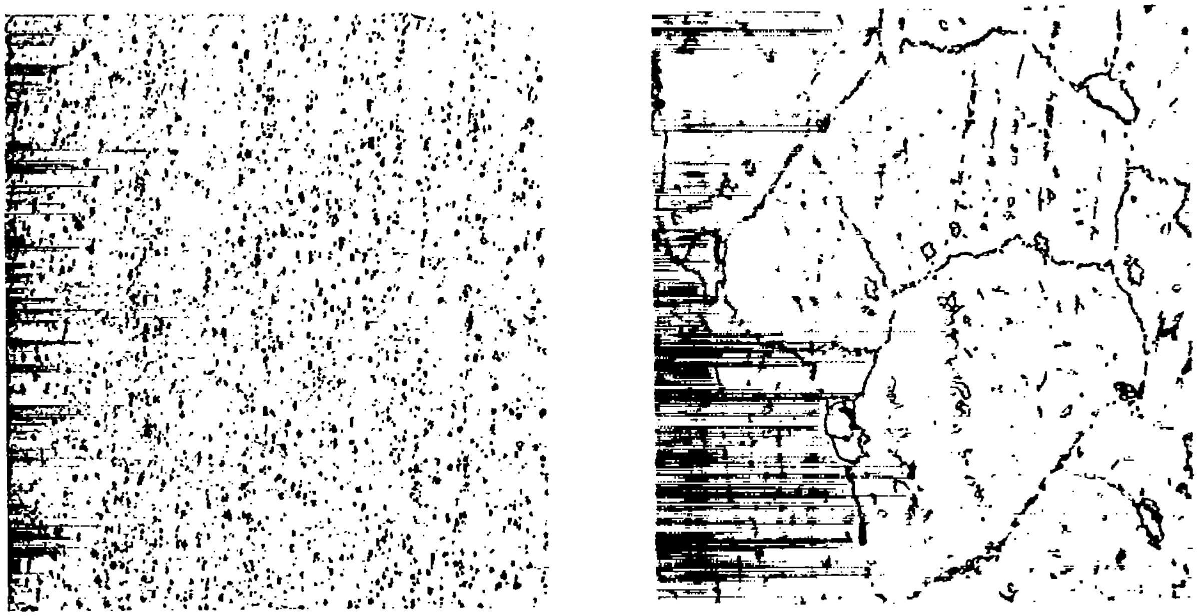
(c) 31 overheats to 1,900° F. Rupture time, 368.5 hours.



X100 X1000

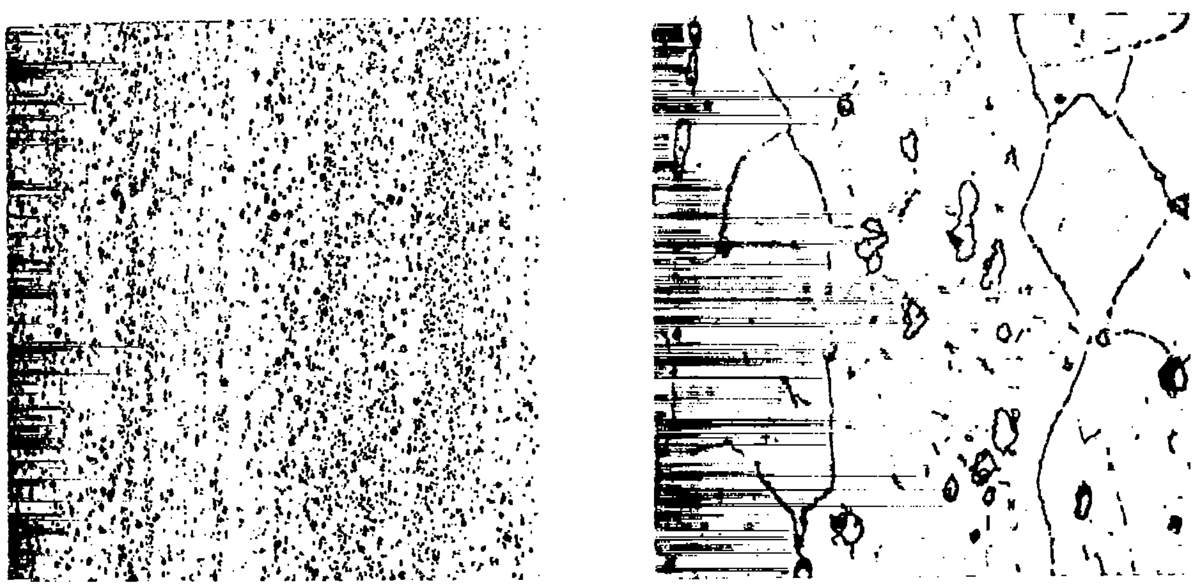
(d) 24 overheats to 2,000° F. Rupture time, 287.8 hours.

Figure 20.- Concluded.



X100 X1000

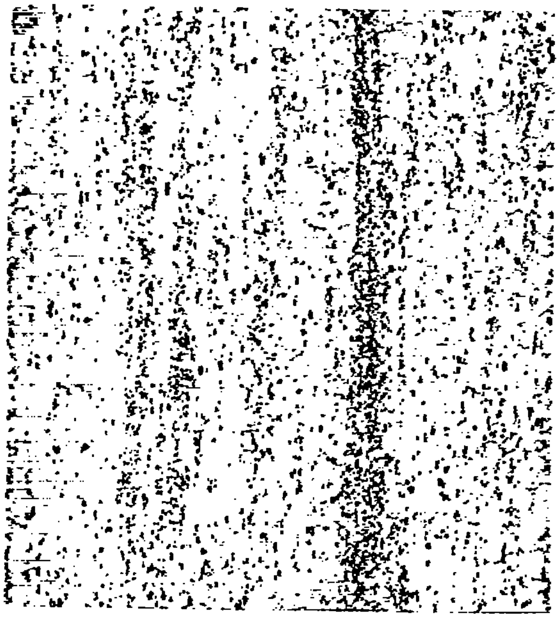
(a) Three cycles of overheats to 1,900° F. Sample run to rupture at 677.2 hours at 1,500° F and 16,200-psi stress.



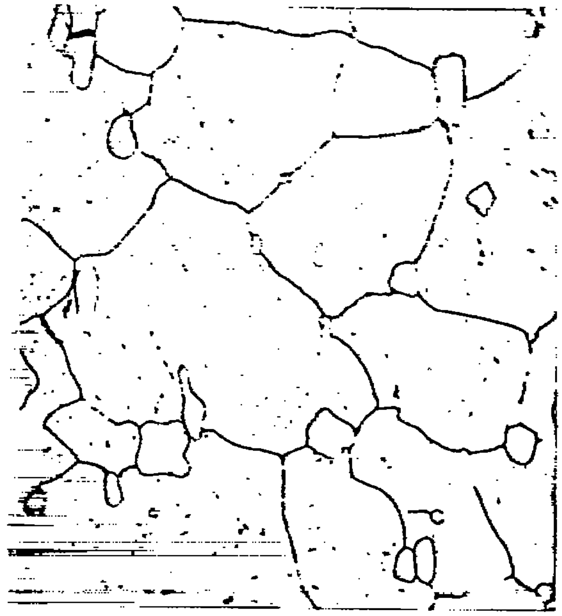
X100 X1000

(b) Fifteen cycles of overheats to 1,900° F. Sample run to rupture at 511.2 hours at 1,500° F and 16,200-psi stress.

Figure 21.- Effect of limited cyclic overheating to 1,900° and 2,000° F on microstructure of specimens tested at 1,500° F and 16,200 psi. Stress removed during 2-minute overheat cycles every 12 hours for indicated number of cycles.



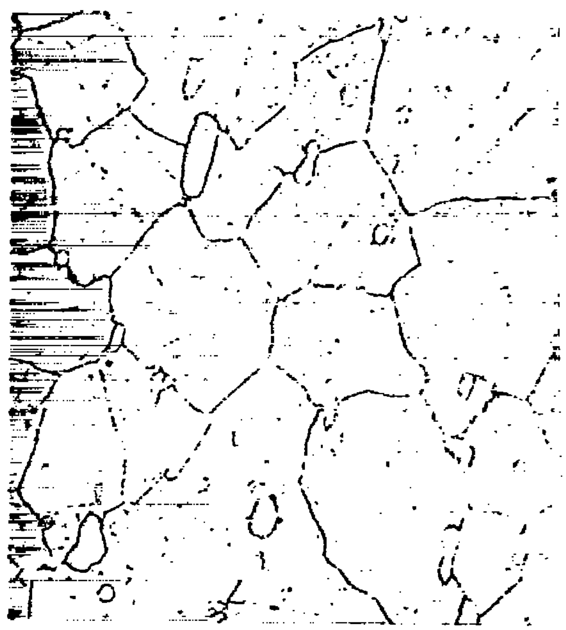
X100



X1000

(c) Five cycles of overheats to 1,900° F. Sample run 50 hours, then cooled to room temperature.

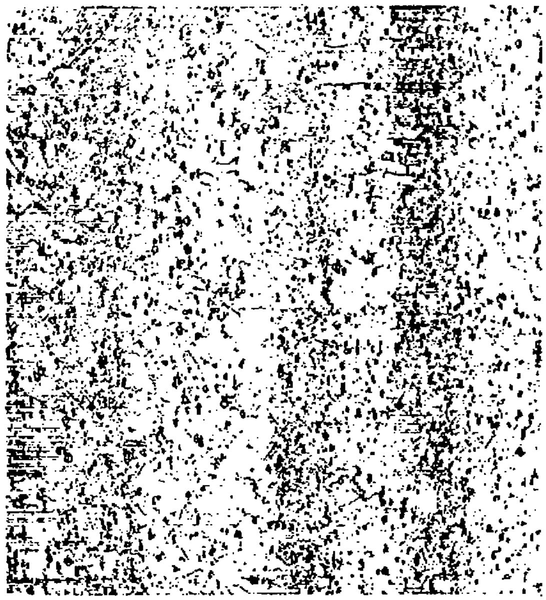
X100



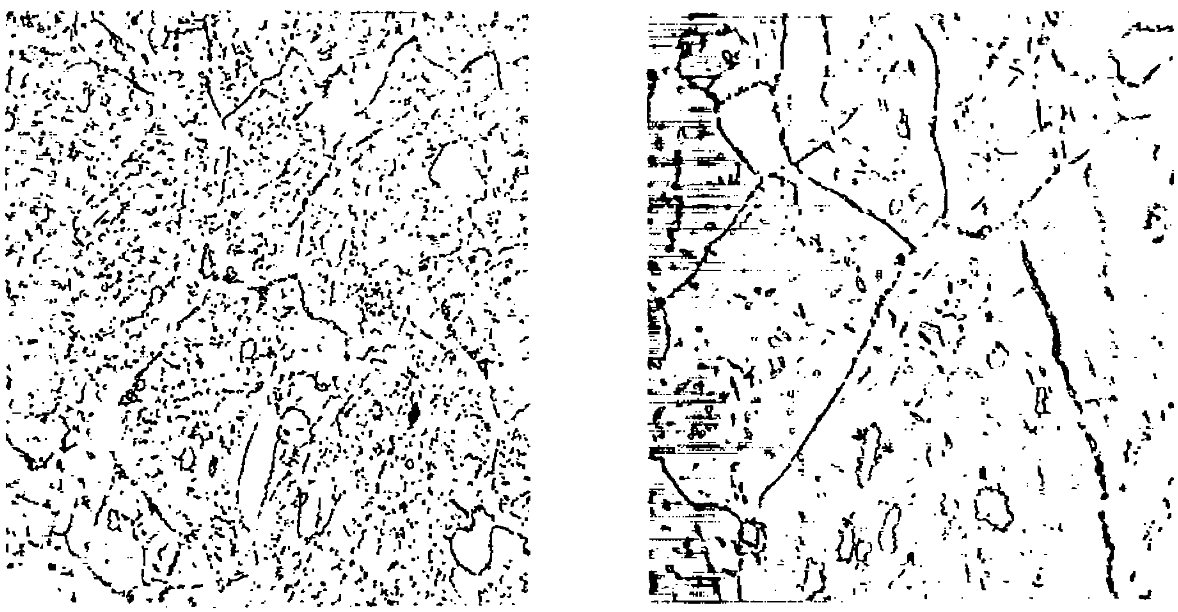
X1000

(d) Twenty cycles of overheats to 1,900° F. Sample run 240 hours, then cooled to room temperature.

Figure 21.- Continued.



X100



X1000

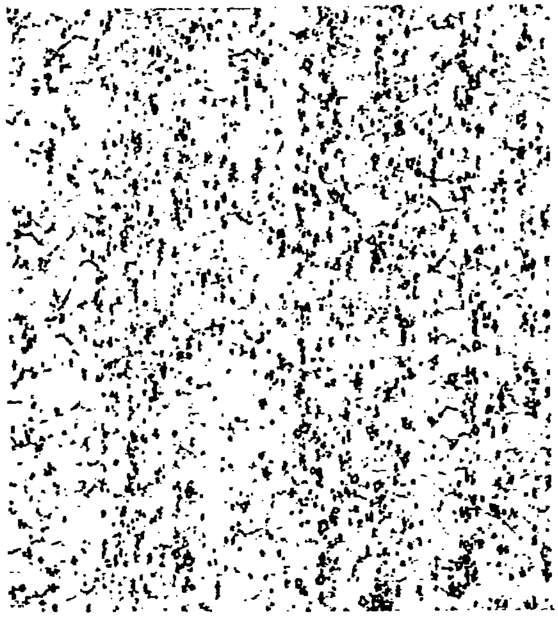
Dark area from photomicrograph above

X1000

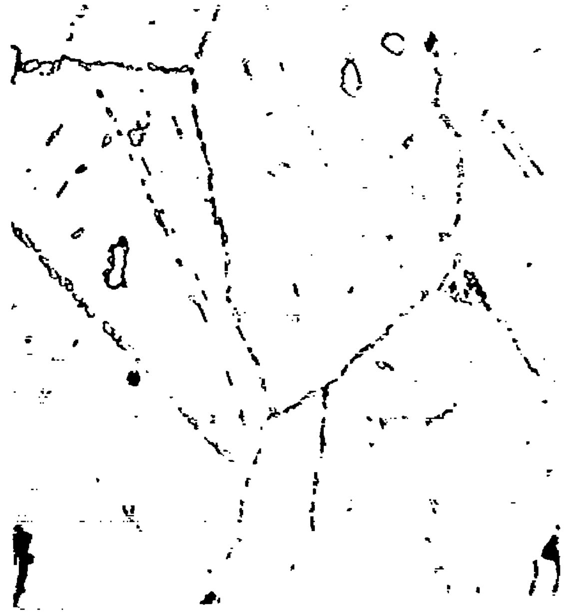
Light area from photomicrograph above

(e) One cycle of overheat at 2,000° F. Sample run to rupture at 438.6 hours at 1,500° F and 16,200-psi stress.

Figure 21.- Continued.



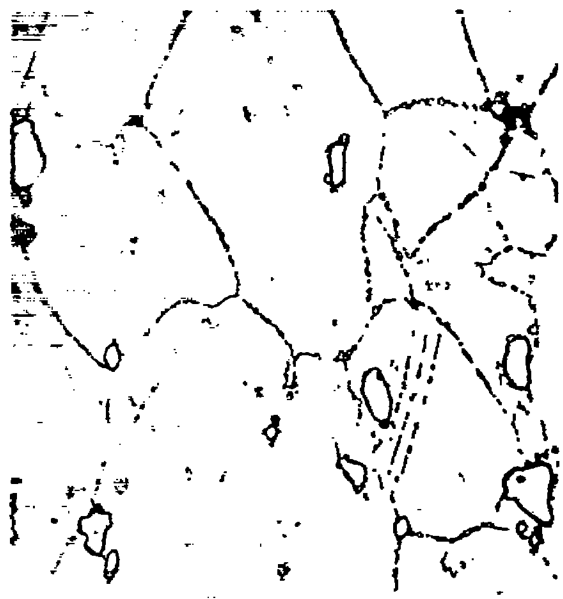
X100



X1000

(f) Two cycles of overheats at 2,000° F. Sample run to rupture at 349.1 hours at 1,500° F and 16,200-psi stress.

X100

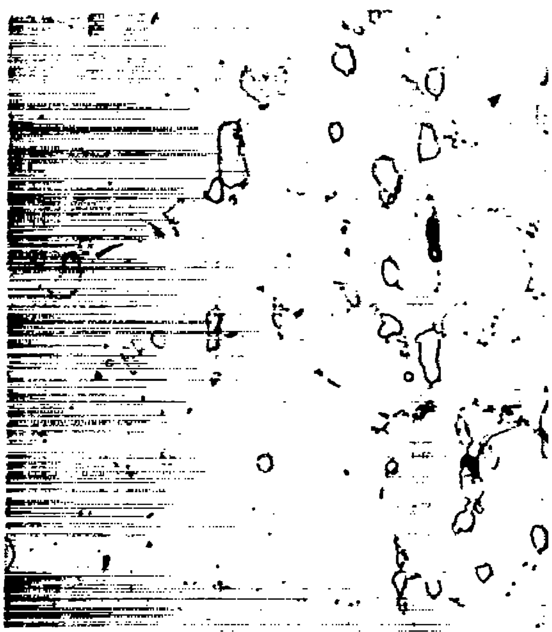


X1000

(g) Ten cycles of overheats at 2,000° F. Sample run to rupture at 291 hours at 1,500° F and 16,200-psi stress.

Figure 21.- Concluded.

X100



X1000

Figure 22.- Effect on microstructure of rupture-testing at 2,000° F and 6,000 psi. Sample ruptured in 0.92 hour.

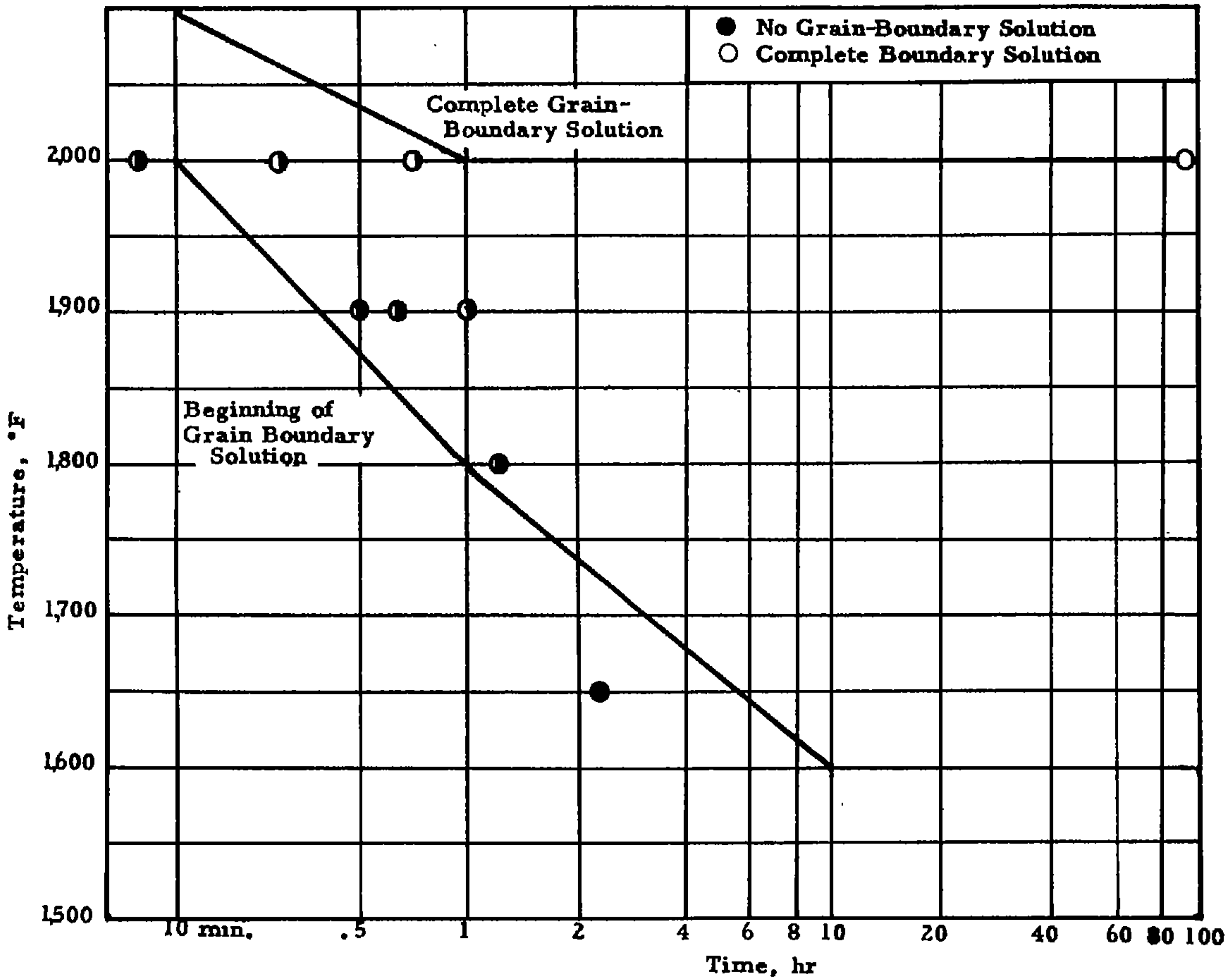


Figure 23.- Extent of grain-boundary solution in fractured overheat specimens compared with lines taken from figure by General Electric Co. showing beginning and completion of grain-boundary solution as a function of time at temperature.

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